

Thermoplastic/metal composite bonding in the field of transport

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Abstract

This project deals about the best process to bond different thermoplastic composites with metals. The composites are Carbon/PEEK, Carbon/PPS and Glass/Pa12; and the metals are aluminum and stainless steel. The three different composites should be bonded once with the aluminum and once with the stainless steel. There will be thus six combinations.

In big projects it is very useful to plan a project with different tools. During the classes in the beginning of the project it was taught which kind of tools exists and what methods are good to plan a project. The first part of the report hence deals with the planning and project management of this project. It is about the scope of the project; a 'work breakdown structure', where all the tasks are listed; a Gantt chart which includes all different tasks, resources and time planning, and different methods to monitor a project.

The next chapter 'state of the art' is about information of composites and metals and about the different surface treatments. Surface treatments are important to enable the adhesive to spread on the surface and to achieve a good stability. In the chapter, 'surface energy', it is explained what exactly it is and how you can measure it with the 'drop test'. Following the different adhesive groups are listed and the adhesion theory explains why bonding is, at all, possible.

The next and almost last chapter deals about the description of the whole bonding process. The three steps, a bonding process includes, are the surface treatment, the adhesive application and finally the joint assembling. The first step is therefore to find the best surface treatment by doing drop tests and shear strength test with the same adhesive for all treatments. Abrasion is the best treatment for the metals and flame for the composites. The second step is to select three different adhesives to find out the best adhesives. This was done by bonding one material among themselves and making shear strength test. So the best adhesive for the combinations PEEK-aluminum, PEEK-stainless steel and PPS-aluminum is the 'DP490'. For the PPS-stainless steel combination, it is 'Duralco 4524'.

Finally the required shear stress were achieved with 15,40 MPa for PEEK-aluminum, 22,40 MPa for PEEK-stainless steel, 23,24 MPa for PPS-aluminum and 16,01 MPa for PPS-stainless steel.

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1 INTRODUCTION

The European Project Semester promotes international student teamwork. Cross cultural and multidisciplinary project groups of three to six international students work together to carry out an integrated-design and business project.

During the 16 weeks of the EPS, students participate in intensive and project-supportive courses, technical courses and English and French lessons. During the first two weeks simultaneously the project groups start to discuss and negotiate their proposal.

A huge amount of time of the EPS is devoted to international teamwork, which requires a collective effort and a collective performance of specialists with different kinds of expertise. Each international team is working together to achieve a set of shared objectives. A major part of the importance of the project is derived from the team's own planning and navigation of the project process from beginning to end. The focus is on the overall product or the project realization process, rather than on any specific science or skill.

The aims of the industrial team-based design project can be described as follows:

1. To train students in teamwork and emphasize real life situations
2. To demonstrate the ability to use modern design tools and techniques
3. To demonstrate the ability to plan and run a team based project
4. To show the ability to communicate clearly in writing (a proper project report) as well as by other means

This project is commissioned by Technacol and the purpose of the project, which can be described as an internal project, following recurrent queries from industry, is to better understand composite materials and how they can be assembled with metals. The combinations that will be studied are:

| | | |
|-------------|-----|-----------------|
| Carbon/PEEK | <-> | Stainless steel |
| Carbon/PEEK | <-> | Aluminum |
| Carbon/PPS | <-> | Stainless steel |
| Carbon/PPS | <-> | Aluminum |
| Glass/Pa12 | <-> | Stainless steel |
| Glass/Pa12 | <-> | Aluminum |

The aim is to find the best bonding process for each combination, which means to find the best surface treatment for each material and the best adhesive to bond the combinations.

To do this, in a correct manner, the following topics will be treated in this report:

- Project Management
- Composites and metals
- Surface preparation
- Adhesives
- Bonding processes

1.1 What is Technacol?

Technacol is a Regional Centre for Innovation and Technology Transfer Specialized in Adhesive Bonding in France. Its mission is to meet the needs of industry in making use of reliable and powerful solutions. Indeed, a good mastery of bonding, including the structural bonding (one of the 100 key technologies identified by the Ministry of Industry) may allow:

- Productivity gains interesting (automation files ...)
- Reduced costs (less tooling, labor)
- Relief structures (use of lighter materials),
- A design led (freedom in shapes and materials).

Thus, for over 20 years, they study the bonded systems, from design to industrialization, on behalf of any industrial sector. Their preferred areas of work are:

- Characterisation and design of glued products (design, choice materials and surface treatments ...),
- The validation aging of adhesive-based mechanical or thermo mechanical
- The implementation and industrialization of the assembly process

Finally, Technacol enjoys total independence against manufacturers and suppliers of adhesives and equipment. This feature ensures complete objectivity in the results given to their customers.

1.1.1 The structure

TECHNACOL is an equipped associative structure. It also makes use of the Research Laboratories belonging to the Ecole Nationale d'Ingénieurs (National Engineering School) in Tarbes, in the South of France. Six people are employed and work in collaboration with the Engineering school professors.



Figure 1-1: National Engineering School in Tarbes

1.1.2 *Fields of activity*

- Adhesive Bonding all materials- from Design to Manufacture
- Calculation and dimensioning of adhesive bonds
- Surface preparation selection
- Choice of adhesives and their processing
- Bonding characterization (ageing, mechanical testing, non-destructive testing, ...)
- Definition of control tests and procedures
- Development of pilot units
- Pre-series, prototypes



Figure 1-2: Some of Technacols laboratories equipments

2 MANAGEMENT

During this semester the project management has been one of the most important issues. The ENIT University offers worthy classes to learn what is necessary to have a powerful project management and they also provides the tools to do it successfully.

What is Project Management?

Project management is the application of knowledge skills, tools and methods to a project with a view to achieve and exceed the needs and expectations of the project stakeholders, i.e. finding a balance between the competing constraints peculiar to projects.

- Different needs and expectations of stakeholders
- Identified requirements (needs) and unidentified requirements (expectations).
- Scale, quality, time and cost.

What are the Project Management objectives?

The main objectives of the Project Management could be listed by this way:

- Meet client needs and expectations
- Satisfy the project team's hopes and expectations
- Provide the project management team with the information needed to make the decisions which will enable them to honour the terms of the client contract (scope, quality, time, cost...)
- Put together statistical data, incidents plus reliable and reusable results to improve preparation and execution of future projects

To have a successful project there are two main topics that should keep in mind from the very beginning till the end of the project:

- Client satisfaction
- Project team satisfaction

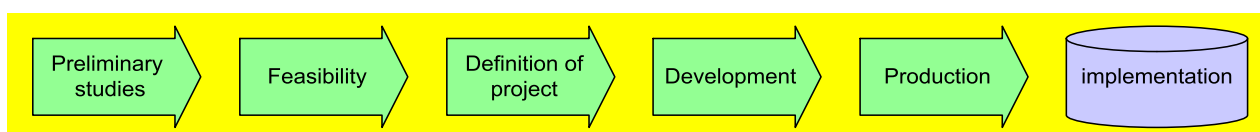


Figure 2-1: Life cycle of a project

Project Management processes

Management of a Project is composed of 5 process types:

1. Initiating:
 - a. Ensure that input data is available
 - b. Get the company to commit to starting the next process
2. Planning:
 - a. Draw up and put into use a workable plan of execution
 - b. Allocate budgets, responsibilities, deadlines, etc.
3. Executing:
 - a. Co-ordinate staff and resources necessary for the execution of the project.
4. Controlling/monitoring:
 - a. Monitor and measure progress.
 - b. Define and employ corrective actions.
5. Closing:
 - a. Formalize completion of the project (prepare administrative documents)
 - b. Build on information acquired during the project
 - c. Assign stakeholders to their new roles

2.1 Scope of the project

A project scope is the work that has to be accomplished to deliver a product, service or result with the specified features and functions. Following pages explain the scope of this project.

2.1.1 Objectives

The aim of the project is to find the best bonding process to assemble 3 different thermoplastic matrix composites (TPMC) with 2 different metals.

- TPMC = Carbon/ PEEK, Carbon/PPS and Glass/Pa12
- Metals = Stainless steel (316L) and aluminum (2017)

| | | |
|-------------|-----|-----------------|
| Carbon/PEEK | <-> | Stainless Steel |
| Carbon/PEEK | <-> | Aluminum |
| Carbon/PPS | <-> | Stainless Steel |
| Carbon/PPS | <-> | Aluminum |
| Glass/Pa12 | <-> | Stainless Steel |
| Glass/Pa12 | <-> | Aluminum |

With a limited budget, we must find the best surface treatment for each material and find the best adhesive for each combination. The bonded samples must also achieve a minimum of 15-20 MPa at a shear strength test. As the outcome of this project will be used by nautical and automotive development, it is also important that the bonded material have a good impact and salt spray resistance and also a good heat and vibration resistance.

Since the project has to be finished on 26 of January 2011, an important key indicator is to start the stability tests before 16 of December 2010.

2.1.2 Clients and Deliverables

One very important thing in all projects is to identify the clients and the involved stakeholders. The results of this project will be used for nautical and automotive development so these companies are the end customers. But the clients that we are responsible for and have deliverables for are ENIT and Technacol.

- Philippe Clermont (ENIT's professor) – all management reviews
- Gladys Chartier (Technacol) – all technical reviews

These are the two main clients that we need to collaborate with and get approval from if we have to change anything regarding to the project. They are also the ones that can stop the project and need to have progress reports on the project. You can find the Customers Structure Level (CSL) in the next page with their deliverables.

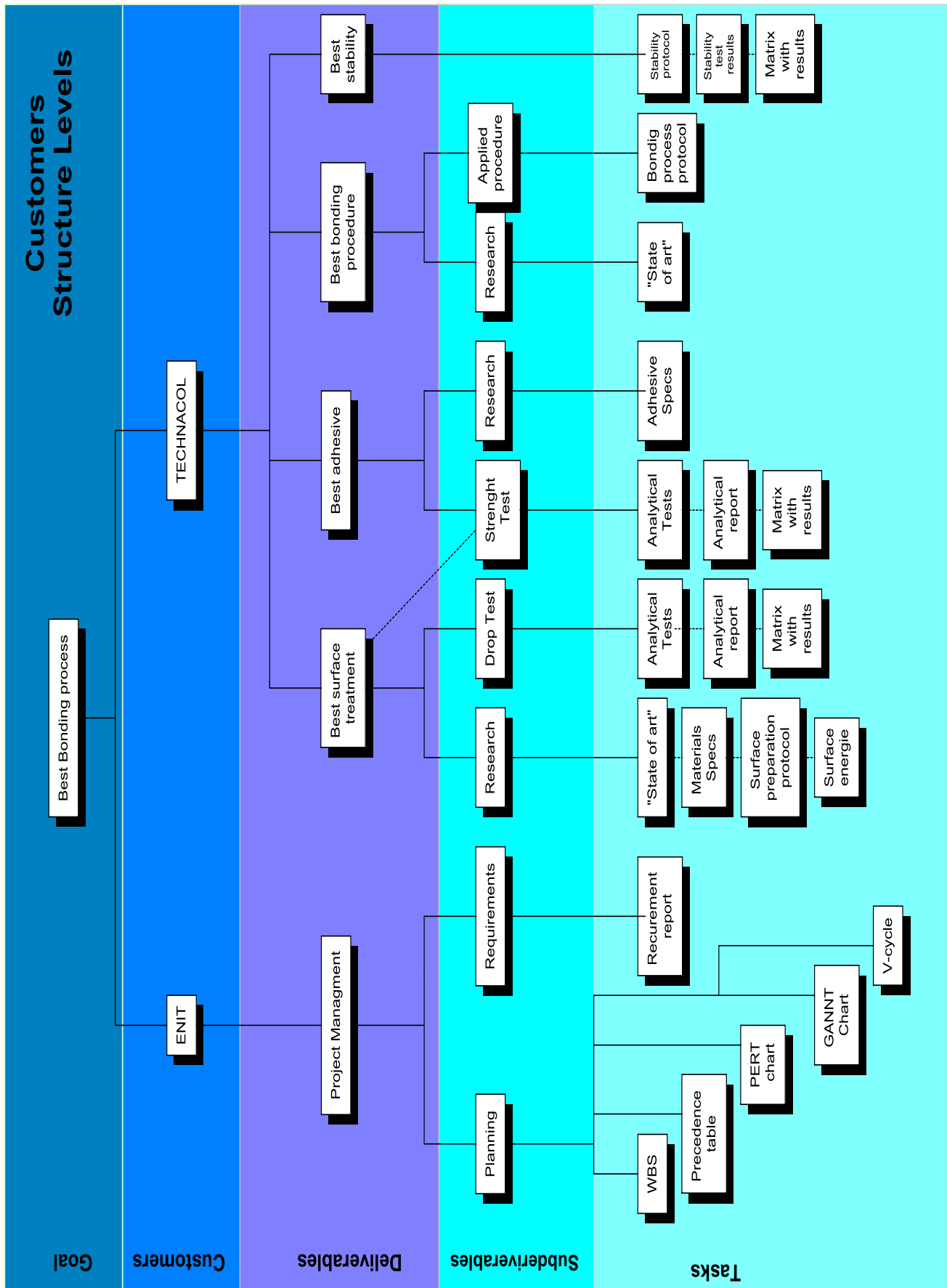
The deliverables we have to give our clients is all the work that we do during these 3 project months. That includes the management and the technical parts.

The management deliverables are

- The Scope of the project
 - Objectives
 - Clients
 - Deliverables
 - Milestones
 - Technical requirements
 - Limits and exclusions
 - Risks
 - Review the content with the clients
- The project planning
 - WBS
 - Gantt chart
 - Precedence table
 - Temporal networks
 - V cycle
- Intermediate project review presentation and report
- Final presentation and report

The technical deliverables are

- Result of the best surface treatment for each material
- Result of the best adhesive for each combination
- Results from the shear strength tests
- Results from the stability tests
- Final report and presentation
- Matrix of all materials and best bonding solution



2.1.3 Milestones

There are four important milestones that are listed below. All of the milestones are both technical and management milestones except the stability test that is only a technical milestone.

- Present the planning – the first version of our planning
Deadline: 05/11/2010
- Intermediate review – a report and presentation of all our previous work
Deadline: 08/12/2010
- Stability test – start the stability tests before the Christmas holidays
Deadline: 16/12/2010
- Final review – a final report and presentation of the project
Deadline: 26/01/2011

2.1.4 Requirement document

The requirement document is an important part of the project. The document ensures a result with client expectations and should therefore be signed by all parties. The requirement document is different for the technical part and the management part. See appendix 1 for the requirement document.

2.1.5 Risks

It is very important to identify and manage the risks in an early stage of a project. There are many different risks that can give bad impact on the project and also in some cases delay the whole project. The risks we have in our project are shown below by the 5 M's:

- **Mission**
 - Not enough raw material – composites, metals, adhesive, chemicals or sandpaper
 - Delayed delivers – shear strength machine, plasma machine, sandblasting samples
 - Unavailable equipments – shear strength machine, corona machine, flame machine, drop test machine, stability test machines, oven and heater
 - Non-functioning machines
- **Man**
 - Human resources – cannot afford to lose manpower, doing tasks bad and not in time, communication problems
- **Media**
 - Communication devices – internet access, telephone availability
- **Management**
 - Deadlines
 - Team communication

2.2 WBS – Work breakdown structure

The WBS or Work breakdown structure is a technique by which we define and quantify the work to be done throughout the project.

It is a thought process through which one tries to organize the project. It is quite similar to the traditional organization of a company, which has a Director, Assistant Directors, Heads of departments, heads of offices, etc.

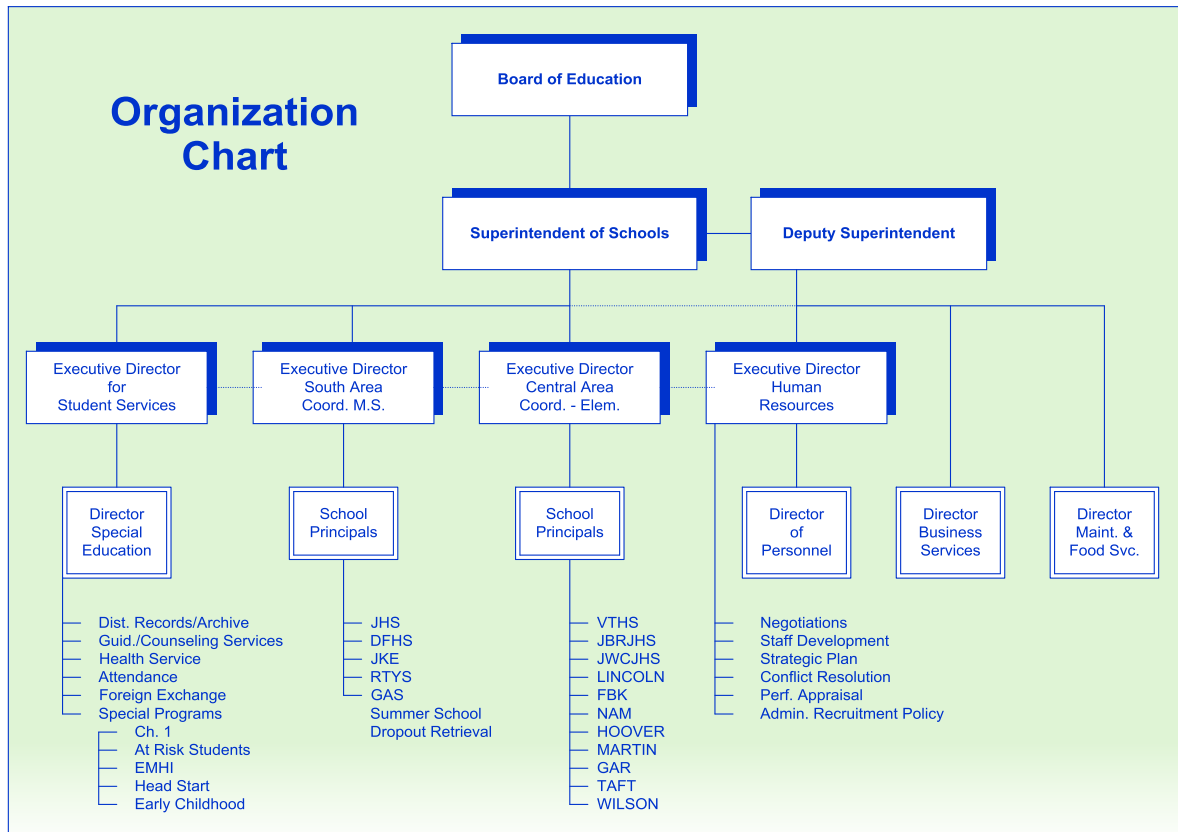


Figure 2-2: Example of a traditional organization

In order to establish the project WBS, we have to organize our ideas about what we want to do in the project, which means to set the title of our research project, according to the goal we want to reach.

Next, we must think about the major areas of work in which the project can be divided, that will form the work packages to be developed to achieve the goal.

To achieve this process in the development of the WBS, we can successfully use a brainstorming technique in a workgroup.

Following to that, each of these work packages can also be examined through a brainstorm and develop a list of constituent activities of each work package. At the same time, these activities can be subdivided to achieve the necessary breakdown. The level of detail required for the project will be determined by the complexity and size of the project. But you can consider the following recommendations to see if you have the level of detail required:

5. The work packages must be independent of each other.
6. Activities at the higher level must be measurable, that means, you can set an estimated execution time and necessary resources to carry out accurately. This part of the process should be done by the more experienced investigator.
7. It is also necessary that each activity is reflected in something "tangible", such as: developments of a drawing or plan, conducting an experiment, buy a computer, write a chapter of the thesis, etc. So that it can be also quantified its actual progress in the implementation stage, monitoring and control of the project.

An interesting concept mentioned now in the coding of activities, it is convenient to use a code to identify activities, indicating the different levels of details, for example:

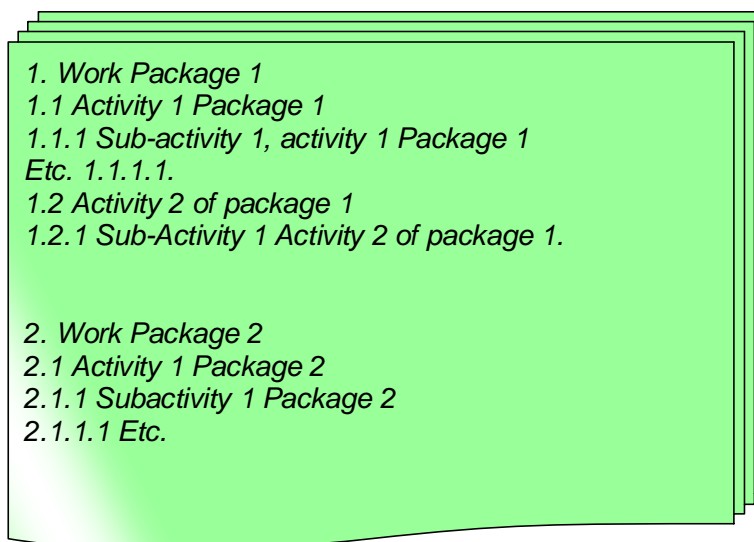


Figure 2-3: Example of identifying work packages and activities

We suggest using this method of encoding levels of detail, since this same method automatically uses MS-Project, which will be used later.

As shown, it is intended that the WBS includes all the activities that will be developed during the project and also left out those that are not concern.

Once the WBS is finished we will have defined the amount of work to do, entirely organized by area or specialty packages, we also have an identifier code through the list of activities.

Each work package enables:

- Definition of the work
- Calculation of the time required to complete one package (how much time?)

- Calculation of resources needed to carry out work package (who?)
- Setting of budget (how much money?)
- Identification of progress indicators (control)

The WBS allows “defining from the general to the particular work in the planning stage and measure progress and resources from the particular to the general during the process of project monitoring and controlling.

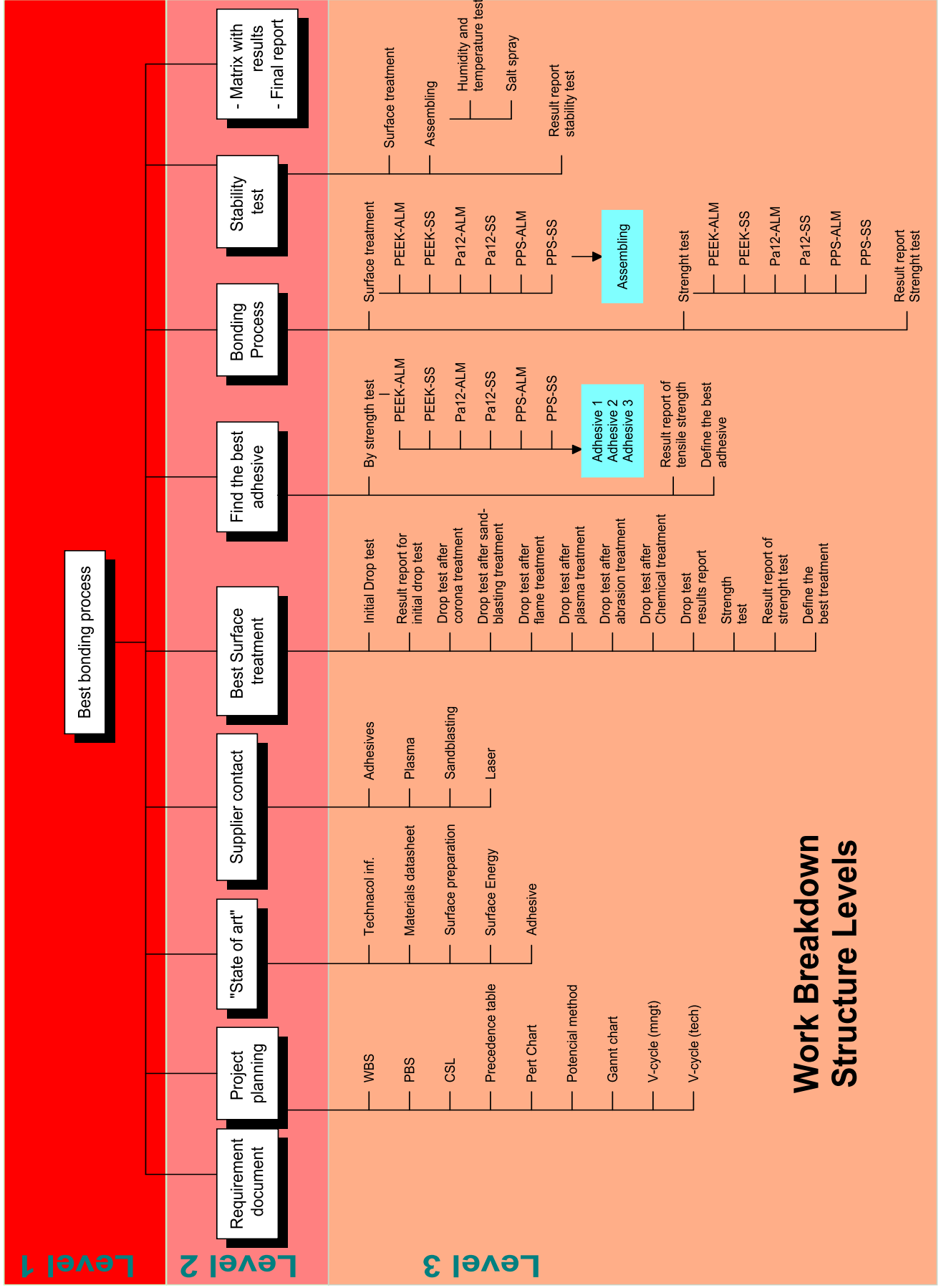
Summarizing we could say that the objective of the WBS is “convert a list of deliverables into a list of tasks”, that’s difficult but this is the key element in project planning.

The benefits of the WBS are:

- Enables identification of 90-95% of tasks to be undertaken
- Breakdown of overall work into a smaller elements
 - easier to handle
 - run by one local manager
 - retains overall coherence
- Facilitates progress measurement
- Applicable to more than 90% of projects, whatever their size or field.

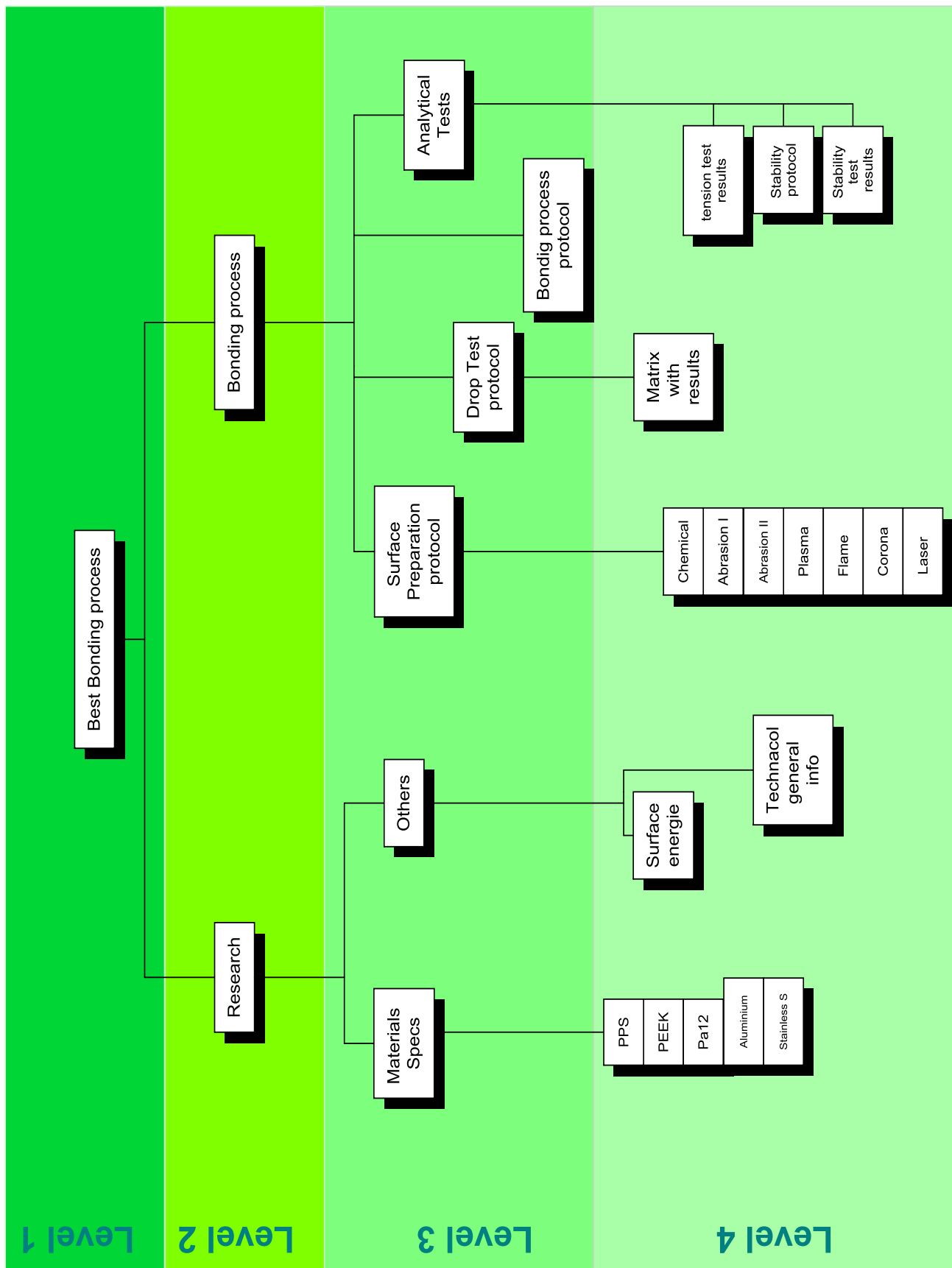
In the next pages you will be able to find the WBS of our project. It has been divided in three different levels, according to the WBS above description.

You also find the PBS, Product Breakdown Structure, in section 2.3 that has the same function of the WBS and the same rules but it is more focused on the product part of the project.



Work Breakdown Structure Levels

2.3 PBS – Product breakdown structure



2.4 Project planning

Project planning is a part of project management, which relates to the use of schedules such as Gantt charts or PERT charts to plan and subsequently report progress within the project environment.

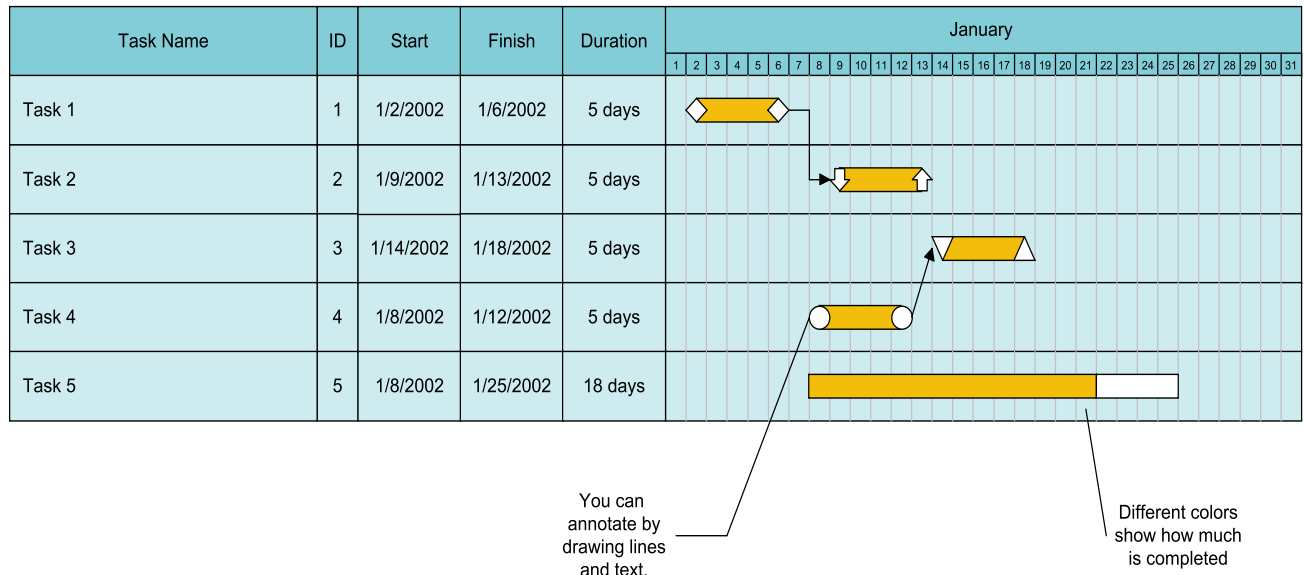


Figure 2-4: Example of a Gantt chart

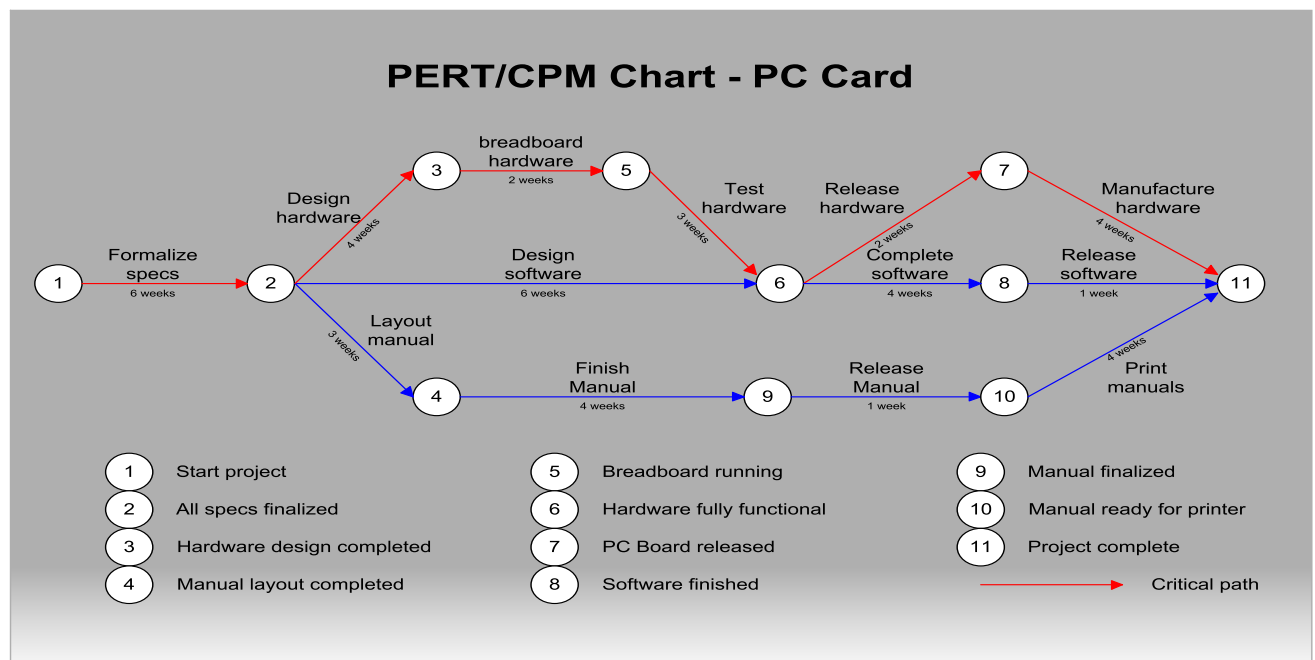


Figure 2-5: Example of a Pert chart

Initially, the project scope is defined and the appropriate methods for completing the project are determined. Following this step, the durations for the various tasks necessary to complete the work are listed and grouped into a work breakdown structure (WBS).

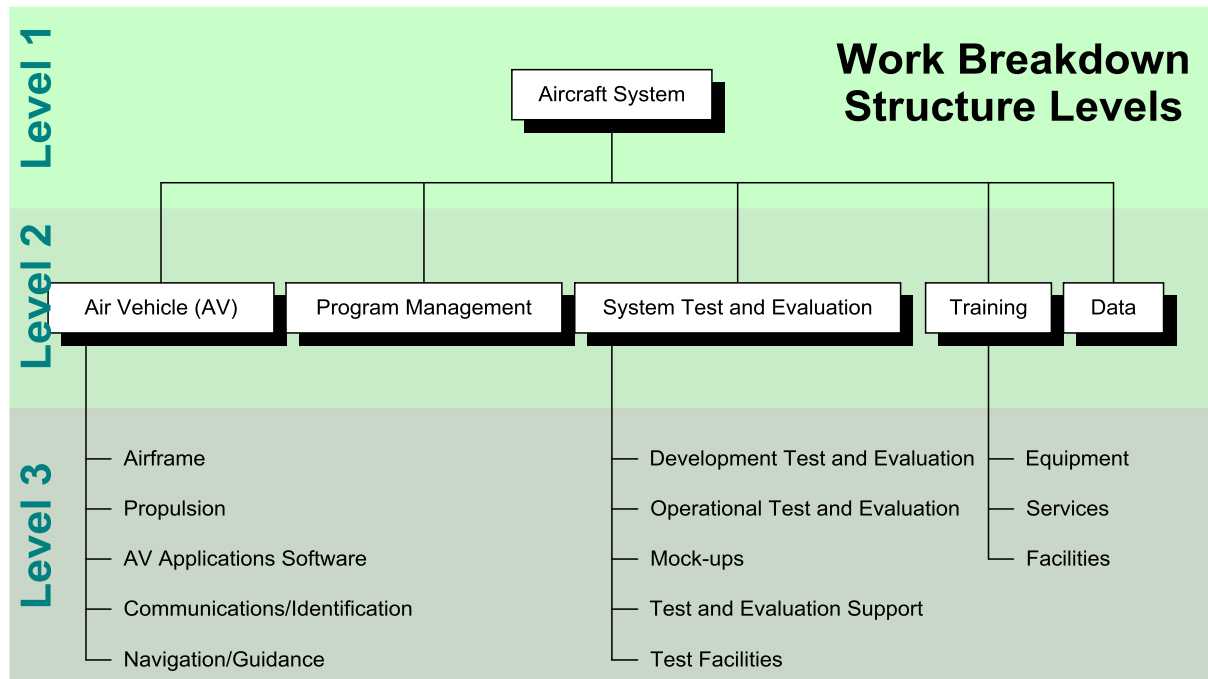


Figure 2-6: Example of a WBS

The logical dependencies between tasks are defined using an activity network diagram that enables identification of theoretical. Float or slack time in the schedule can be calculated using project management software. Then the necessary resources can be estimated and costs for each activity can be allocated to each resource, giving the total project cost. At this stage, the project plan may be optimized to achieve the appropriate balance between resource usage and project duration to comply with the project objectives.

Once established and agreed, the plan becomes what is known as the baseline. Progress will be measured against the baseline throughout the life of the project. Analyzing progress compared to the baseline is known as earned value management.

The inputs of the project planning phase include Project Charter and the Concept Proposal. The outputs of the Project Planning phase include the Project Requirements, the Project Schedule, and the Project Management Plan.

2.4.1 Precedence table

The Precedence table is a Project Planning tool that presents all project tasks in a simple and comprehensive way.

This table is the result of industry experts' analysis of resources to be used to keep to specifications.

The Precedence Table for this project is shown below were the entire project tasks have been called down.

| Activity | Tasks/description |
|----------|-------------------------------------|
| A | Define the scope |
| B | Requirement Document |
| C | Project planning |
| C.1 | WBS – precedence table |
| C.2 | Gantt chart |
| D | Preliminary studies |
| D.1 | Basic information about Technacol |
| D.2 | Datasheet and documentation |
| D.2.1 | Carbon PEEK |
| D.2.2 | Carbon PPS |
| D.2.3 | Glass Pa12 |
| D.2.4 | Stainless Steel |
| D.2.5 | Aluminum |
| D.3 | Surface preparation |
| D.3.1 | Possible treatments and description |
| D.4 | Surface Energy |
| D.4.1 | General information |
| D.4.2 | Measurability |
| D.5 | Adhesive |
| D.5.1 | General information |
| D.5.2 | Datasheet of our adhesive selection |
| E | Supplier contact |
| E.1 | Adhesives |
| E.2 | Plasma |
| E.3 | Sandblasting |

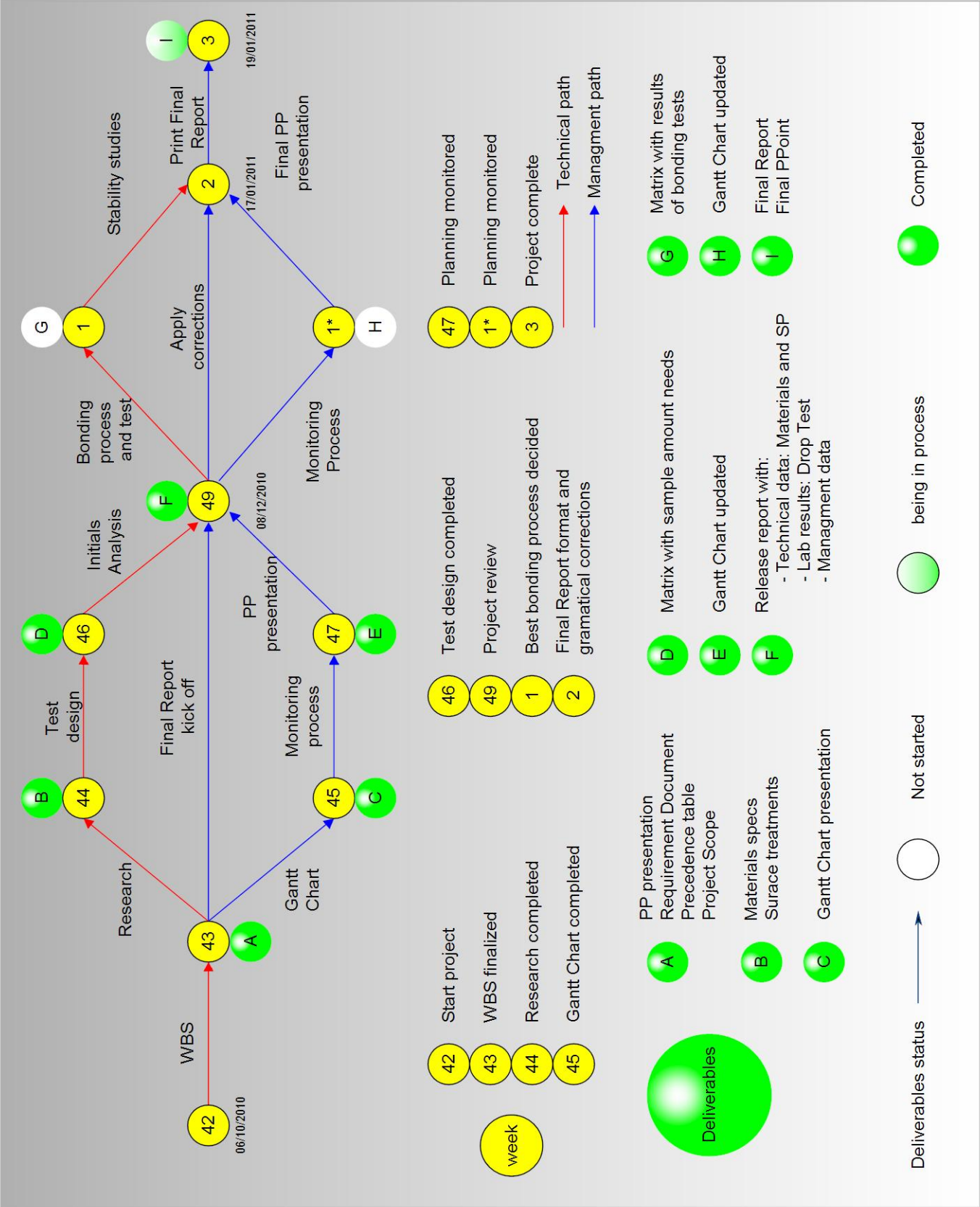
| Activity | Tasks/description |
|----------------|------------------------------------------------|
| E.4 | Laser |
| F | Initial drop test for all materials |
| G | Result report for all initial drop test |
| H | Finding the best surface treatment |
| H.1 | by doing drop test |
| H.1.1 | after corona treatment |
| H.1.1.1 | For PEEK, Pa12, PPS |
| H.1.2 | after abrasion treatment |
| H.1.2.1 | For PEEK, Pa12, PPS |
| H.1.3 | after chemical treatment |
| H.1.3.1 | For aluminum |
| H.1.3.2 | For stainless steel |
| H.1.4 | after flame treatment |
| H.1.4.1 | For PEEK, Pa12, PPS |
| H.1.5 | after plasma treatment |
| H.1.5.1 | For PEEK, Pa12, PPS |
| H.1.6 | after sandblasting treatment |
| H.1.6.1 | For PEEK, Pa12, PPS |
| H.2 | Result report drop test |
| H.3 | by doing shear strength test |
| H.3.1 | for the metals |
| H.3.1.1 | abrasion treatment |
| H.3.1.1.1 | St.Steel-St.Steel |
| H.3.1.1.2 | Aluminum-Aluminum |
| H.3.1.2 | chemical treatment |
| H.3.1.1.1 | St.Steel-St.Steel |
| H.3.1.1.2 | Aluminum-Aluminum |
| H.3.2 | for the composites |
| H.3.2.1 | Treatment 1 |
| H.3.2.1.1 | PEEK-Al. or Steel |
| H.3.2.1.2 | Pa12-Al. or Steel |
| H.3.2.1.3 | PPS-Al. or Steel |
| H.3.2.2 | Treatment 2 |

| Activity | Tasks/description |
|----------------|-------------------------------|
| H.3.2.2.1 | PEEK-Al. or Steel |
| H.3.2.2.2 | Pa12-Al. or Steel |
| H.3.2.2.3 | PPS-Al. or Steel |
| H.3.2.3 | Treatment 3 |
| H.3.2.3.1 | PEEK-Al. or Steel |
| H.3.2.3.2 | Pa12-Al. or Steel |
| H.3.2.3.3 | PPS-Al. or Steel |
| H.4 | Result report shear strength |
| I | Define the best treatment |
| K | Finding the best adhesive |
| K.1 | by shear strength test |
| K.1.1 | PEEK - Aluminum |
| K.1.1.1 | Adhesive 1 |
| K.1.1.2 | Adhesive 2 |
| K.1.1.3 | Adhesive 3 |
| K.1.2 | PEEK - Stainless steel |
| K.1.2.1 | Adhesive 1 |
| K.1.2.2 | Adhesive 2 |
| K.1.2.3 | Adhesive 3 |
| K.1.3 | Pa12 - Aluminum |
| K.1.3.1 | Adhesive 1 |
| K.1.3.2 | Adhesive 2 |
| K.1.3.3 | Adhesive 3 |
| K.1.4 | Pa12 - Stainless steel |
| K.1.4.1 | Adhesive 1 |
| K.1.4.2 | Adhesive 2 |
| K.1.4.3 | Adhesive 3 |
| K.1.5 | PPS - Aluminum |
| K.1.5.1 | Adhesive 1 |
| K.1.5.2 | Adhesive 2 |
| K.1.5.3 | Adhesive 3 |
| K.1.6 | PPS - Stainless steel |
| K.1.6.1 | Adhesive 1 |

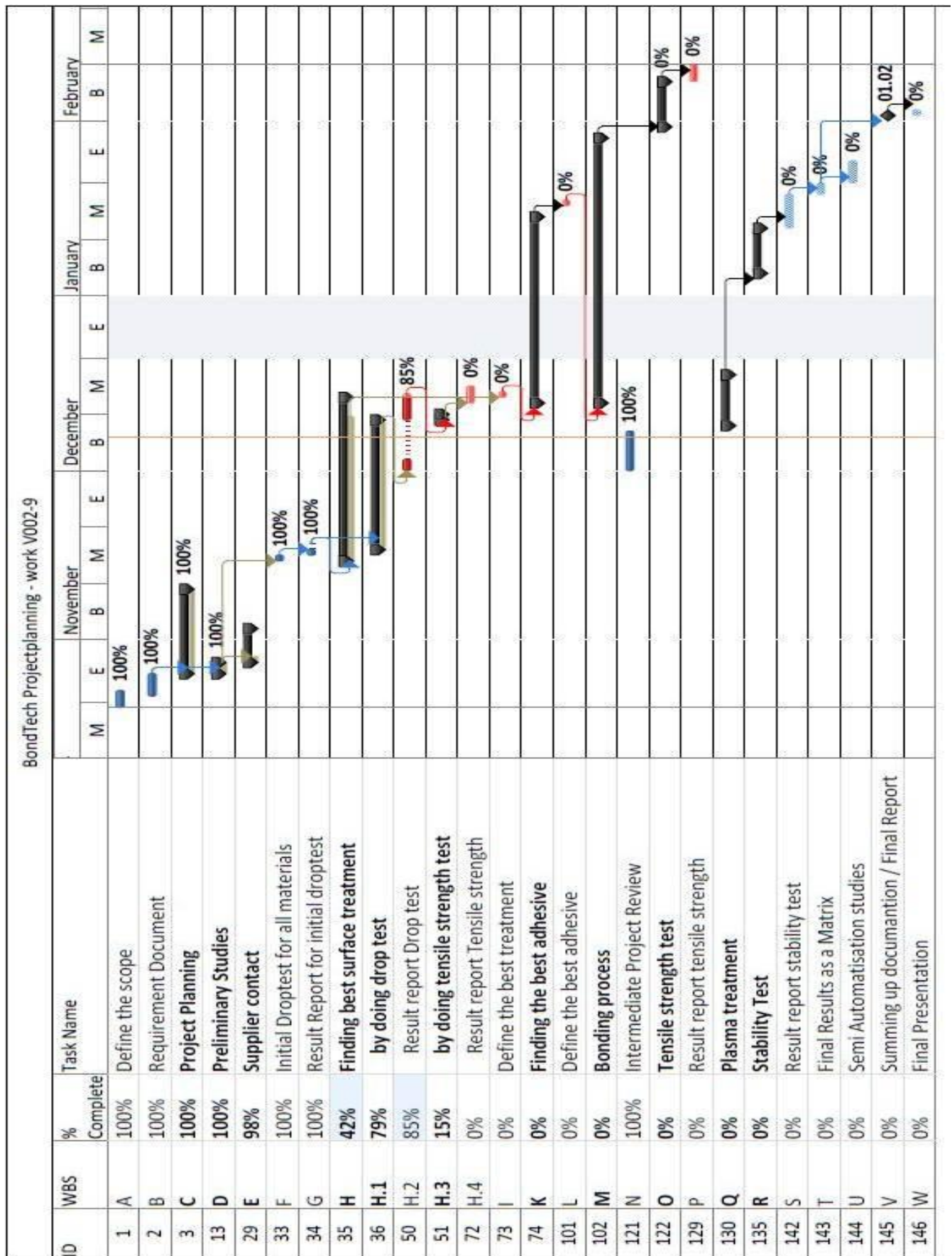
| Activity | Tasks/description |
|----------|-----------------------------|
| K.1.6.2 | Adhesive 2 |
| K.1.6.3 | Adhesive 3 |
| K.2 | Result report adhesive test |
| L | Defining the best adhesive |
| M | Bonding process |
| M.1 | PEEK - Aluminum |
| M.1.1 | Surface treatment |
| M.1.2 | Assembling |
| M.2 | PEEK – Stainless Steel |
| M.2.1 | Surface treatment |
| M.2.2 | Assembling |
| M.3 | PPS - Aluminum |
| M.3.1 | Surface treatment |
| M.3.2 | Assembling |
| M.4 | PPS – Stainless steel |
| M.4.1 | Surface treatment |
| M.4.2 | Assembling |
| M.5 | Pa12 – Aluminum |
| M.5.1 | Surface treatment |
| M.5.2 | Assembling |
| M.6 | Pa12 – Stainless steel |
| M.6.1 | Surface treatment |
| M.6.2 | Assembling |
| N | Intermediate Project Review |
| N.1 | Project report |
| N.2 | PowerPoint presentation |
| O | Shear strength test |
| O.1 | PEEK - Aluminum |
| O.2 | PEEK - Stainless Steel |
| O.3 | PPS - Aluminum |
| O.4 | PPS - Stainless Steel |
| O.5 | Pa12 - Aluminum |
| O.6 | Pa12 - Stainless Steel |

| Activity | Tasks/description |
|----------|------------------------------------------------------------------|
| P | Result report shear strength |
| Q | Plasma treatment |
| Q.1 | Drop test for all materials |
| Q.2 | Finding the best surface treatment by shear strength test |
| | St.Steel-St.Steel |
| | Aluminum-Aluminum |
| | PEEK – PEEK |
| | PPS-PPS |
| | Pa12-Pa12 |
| Q.2 | Finding the best adhesive |
| Q.2.1 | Shear strength test |
| Q.3 | Stability test sample preparation |
| R | Stability Test |
| R.1 | Bonding process |
| R.1.1 | Surface treatment |
| R.1.2 | Assembling |
| R.2 | Stability Process |
| R.2.1 | Humidity and temperature test |
| R.2.2 | Salts prey test |
| S | Result report stability test |
| T | Final Results as a Matrix |
| U | Semi Automatisations studies |
| V | Summing up documentations / Final Report |
| W | Final Presentation |

2.4.2 Pert chart



2.4.3 Gantt chart



2.5 Project monitoring

Project monitoring means to keep a careful check of project activities over a period of time. To work to its full potential, any kind of project needs to set out proposals and objectives. Then a monitoring system should be worked out to keep a check on all the various activities, including finances. This will help project staff to know how things are going, as well as giving early warning of possible problems and difficulties.

How can a project be monitored?

1. **Keep it simple**

Remember... monitoring is meant to be a help to good project management and not a burden.

2. **Objectives**

Work out clearly at the beginning the objectives of the project, including a budget of the likely cost.

3. **Plan the activities**

- what needs to be done
- when it should be done
- who will be involved in doing it
- what resources are needed to do it
- how long it will take to do
- how much it will cost.

4. **Monitoring**

Work out the most appropriate way of monitoring the work - again, keep it simple:

- meetings
- diaries
- reports on progress
- accounts, reports on finances.

Monitoring methods

Reports

These do not have to be very long. Their purpose needs to be clear - to report on activities and achievements. Above is an example of the records kept by ASHA in India. They give a clear and helpful record of exactly what has been achieved. They are short and to the point. This kind of report will help them in future planning and would clearly inform the Government or a donor agency of what has taken place.

Diaries

A helpful way of recording information would be to use one side of a note book for example, for daily or weekly plans. Write on the other side what actually happened. Keeping a work diary like this will help you also to evaluate your own work. What are you doing that is most

helpful and brings effective results? Take time to ask people in the community about how they feel.

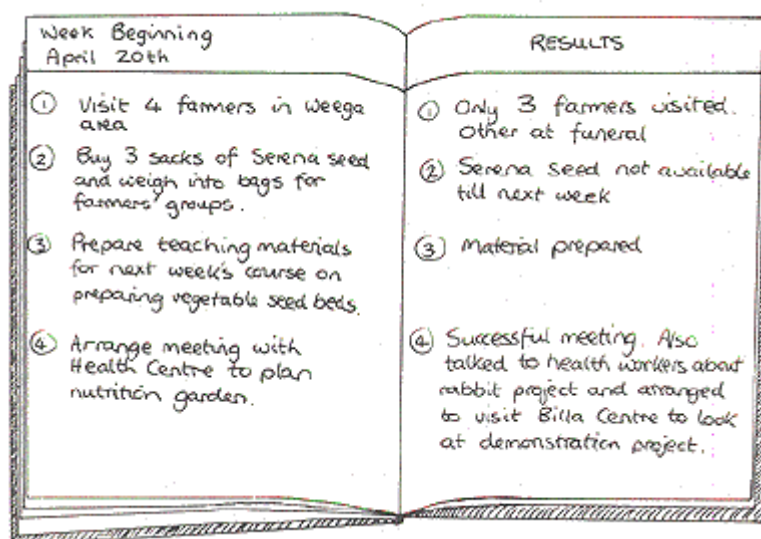


Figure 2-7: Example of a notebook as a diary

Finances

Donor agencies often transfer funds in quarterly or half yearly payments which may not fit in with the planned project expenses. Planning of expenditure may need to take this into account. Careful budgeting and planning will be of great help in this.

Meetings

Confidence and trust are vital. There is a possibility that monitoring may be seen as a way of checking up on people. It must be a joint effort that everyone is involved with in some way or another.

For monitoring to be a useful tool, the information that is collected must be used effectively in all sorts of ways:

- Improve the timing of planned activities.
- Adjust budgets.
- Improve future planning and decision making.
- Indicate where future work is necessary.
- Inform other agencies of activities, to encourage cooperation and publicity.
- Inform funding agencies of progress and future plans.

2.5.1 Tracking Gantt

To do the monitoring process of this project it has been used a powerful tool called Gantt chart. This kind of tool is available on programs as Microsoft Project Planning. As shown below The Gantt Chart diagram express the current status of the project in a specific date and it is easily checked by a simple view.

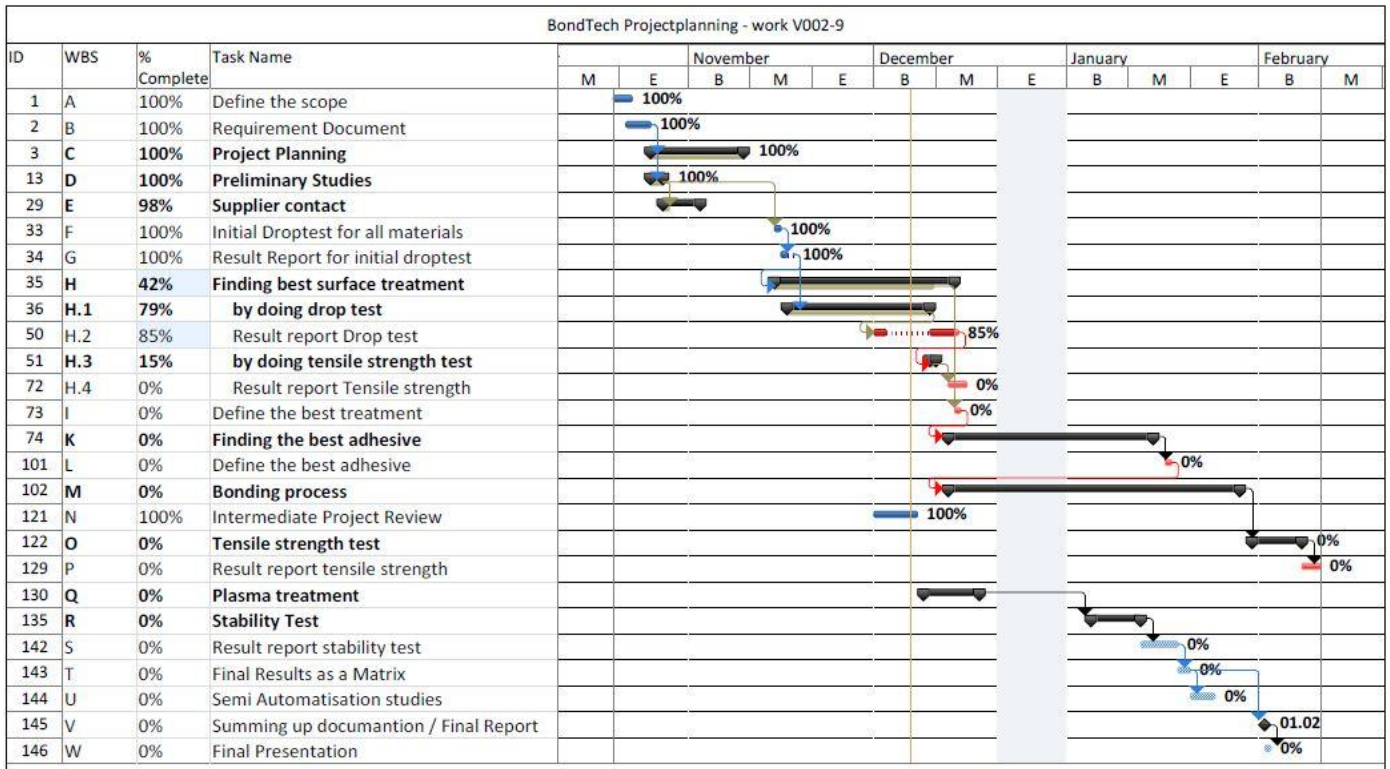


Figure 2-8: Monitoring Gantt chart

As you can see, the status of the project on Dec 6th 2010 is the following one:

- All the research processes have been done.
- Project management and project planning documents has been finished.
- Project monitoring tools has been defined and created.
- Surface treatments are almost finished, plasma one is still pending due that the availability of plasma machine.
- Drop test have been done and evaluated for almost all the surface treatments. There are still pending the plasma and sandblasting drop test. After do all these test it wil be able to define the best surface treatment to apply on every material before assembling process.

The next steps are:

- To check with the shear strength tests if the drop test results give us the right information. Best drop test surface should gives the best shear strength test results

- Once the best surface treatment is defined, the next step is to define the best adhesive. Three of them will be tested for each composite
- After that, the whole assembling process will be ejected and tested with the shear strength test.
- Eventually the Stability tests will be done.

2.5.2 V Cycle

Another way to have an overview of the project and be able to monitor is the V-Cycle.

The V-Cycle is a project development process which may be considered an extension of the waterfall model. Instead of moving down in a linear way, the process steps are bent upwards after the coding phase, to form the typical V shape. The V-Cycle demonstrates the relationships between each phase of the development life cycle and its associated phase of testing. The horizontal and vertical axes represents time or project completeness (left-to-right) and level of abstraction (coarsest-grain abstraction uppermost), respectively.

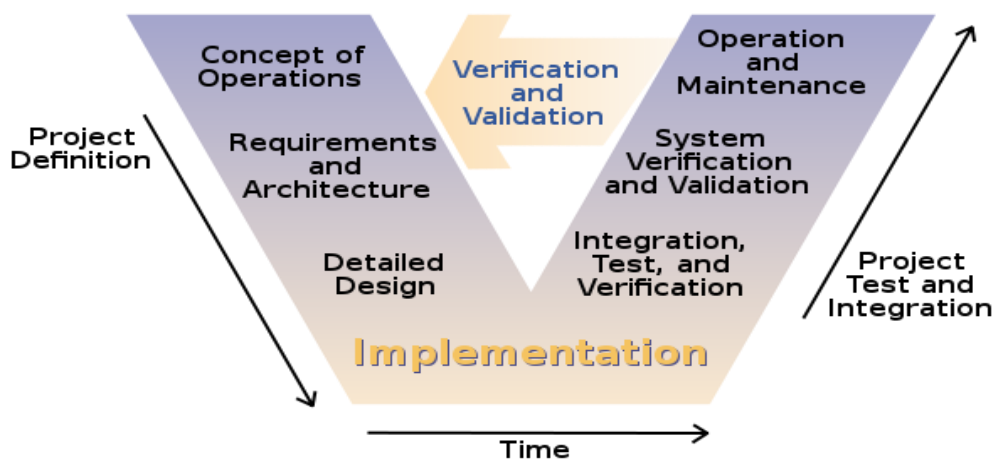


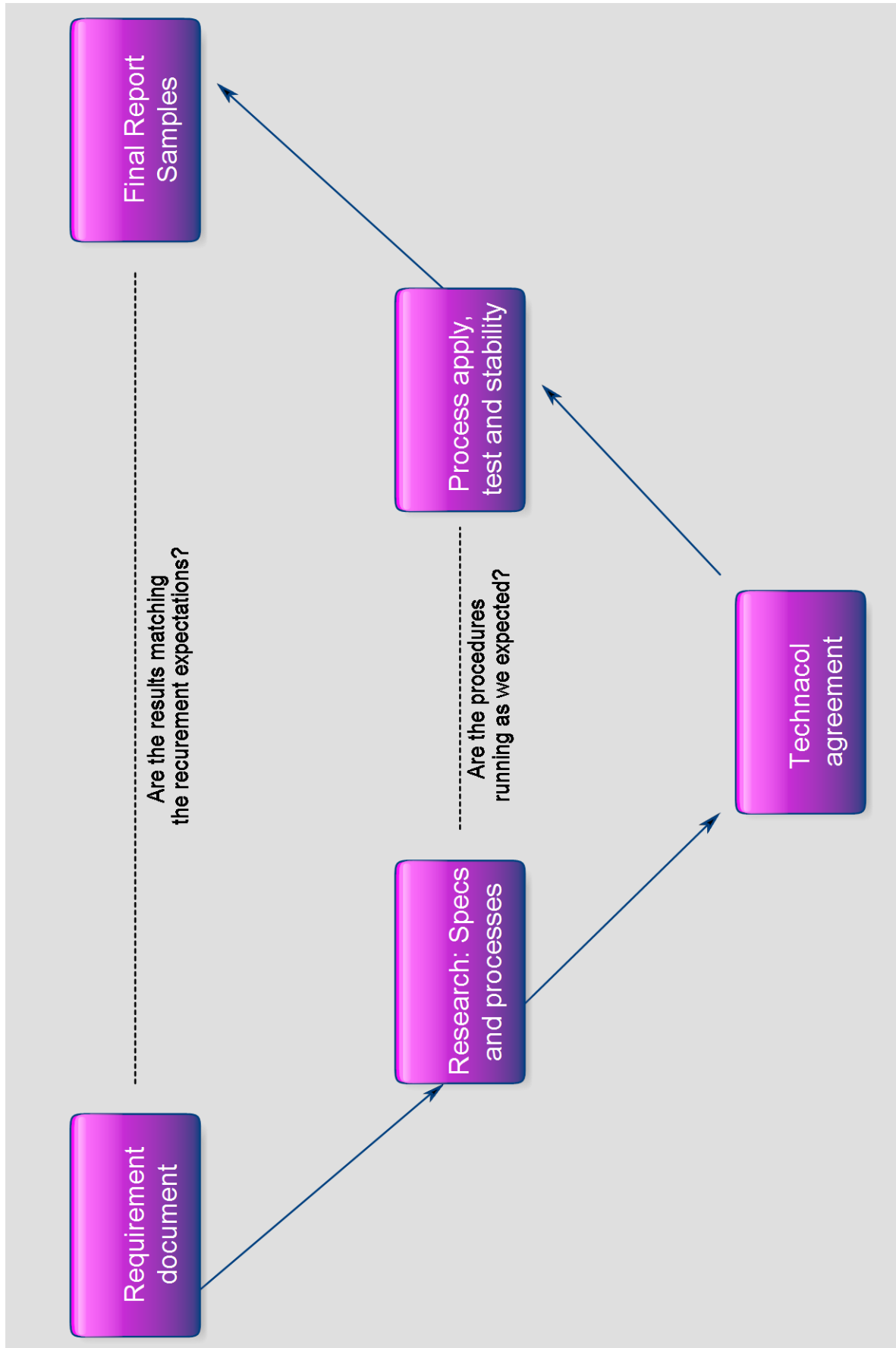
Figure 2-9: Example of a V-Cycle

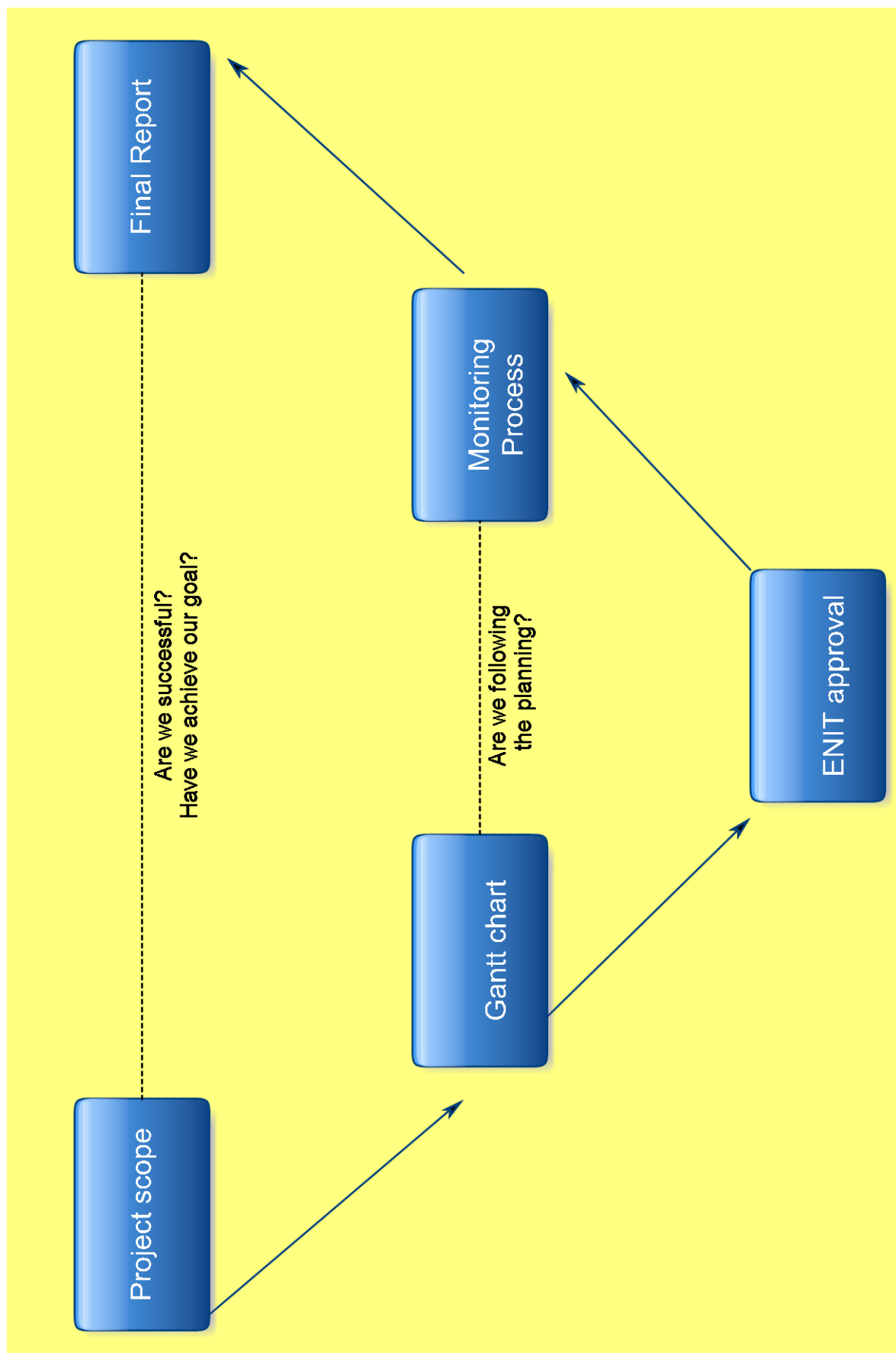
The advantages of use V-Cycle are:

- Overcome situations of the cascade cycle
- Separate functional matters (needs), design and development matters.
- Traceability (global consistency, distribution of updates)
- Errors are detected earlier.

During this project has been created two different V-Cycles were made. One of them is regarding the Project Management needs, wich destination is to satisfy ENIT client.

The other one is more focused on technical deliverables. This one has been created to accomplish the Technacol requirements. Both of them are available in the next two pages.



V cycle – management

3 STATE OF THE ART

3.1 Composites

Composites are materials formed from a mixture of two or more components which, when combined, produce a material with properties or characteristics which are superior to those of the individual materials. Most composites are comprised of two parts, namely the polymer matrix component and the reinforcement component(s). The polymer matrix components are the materials which encapsulate the reinforcing material. The resulting matrixes with reinforcements in place are distributing the load among the reinforcements. Since reinforcements are usually stiffer than the matrix material, they are the primary load-carrying component within the composite. Reinforcements may come in many different forms ranging from fibers, to fabrics, to particles imbedded into the matrix that form the composite.

Composite structures have existed for millions of years in nature. Wood is a wonderful example. Examination of the microstructure of wood reveals the composites material found in nature and suggests that modern composite materials have essentially evolved mimicking structures found in nature. A perfect example of a composite would be concrete.

If you look at the different forms of concrete you will quickly get the idea of how reinforcements work. The cement acts as the matrix, which holds the concrete together, while the sand, gravel, and steel serve as reinforcements. Concrete made with only sand and cement is not nearly as strong as concrete made from cement, sand and gravel, which in turn, is not as strong as concrete reinforced with steel, sand and gravel. The matrix and reinforcement materials of concrete are blended, poured and molded, typically in a form structure. In the generation of parts made with other composite materials, the shape of a composite part is determined by the shape or geometry of the mold, due of other tooling used in the part forming.



Figure 3-1: Long-fiber reinforcing material

There are many different types of composites, including plastic composites. Each plastic resin has its own unique properties, which when combined with different reinforcements create composites with different mechanical and physical properties. Considering the number of plastic polymers in existence today and multiplied that figure by the number of reinforcements available, the number of

potential composite materials is staggering. Of all the available plastic composites, there are two primary categories: thermoset and thermoplastic composites.

In the case of thermoset composites, after application of heat and pressure, thermoset resins undergo a chemical change, which cross-links the molecular structure of the material. Once cured, a thermoset part cannot be remolded.

Thermoplastic materials are not as constrained as thermoset materials and can be recycled and reshaped to create a new part. Thermoplastics that are reinforced with high-strength, high-modulus fibers provide dramatic increases in strength and stiffness, as well as toughness and dimensional stability. The performance gain achieved from thermoplastic materials is often justification that compensates for the increased costs of most thermoplastic materials.



Figure 3-2: Thermoplastic raw material

Composite materials are used in numerous applications across a broad range of industries. Typically, composites are used to replace products made of metal alloys. Composites can offer comparable or better strength than metal parts, while providing a reduction in weight. This is particularly important in industries such as automotive and aerospace, where the use of composite materials results in lighter, faster and more fuel-efficient aircraft and automobiles.

Thermoplastic composites may also be designed to replace wood, fiberglass, concrete and other more traditional materials. The following is a partial list of industries that may have application for the use of long-fiber reinforced structural parts made from thermoplastic composites:

- Aerospace
- Automotive
- Construction
- Home Appliance
- Marine
- Material Handling
- Medical
- Military
- Telecommunications
- Transportation
- Waste Management
- Specialty Products

In general, among other attributes, composite materials are corrosion resistant and offer long fatigue life, which have made them particularly attractive for many manufacturers. The fatigue life refers to the period of time the part will last prior to exhibiting material wear or significant stress. Typically, composites are substituted for conventional materials where there is a desire to reduce the weight of a particular part while providing the required strength and other performance criteria.

Numerous parts, particularly in the aerospace industry, have been and are being made from "advanced" thermoset composite materials, which are very expensive. These types of advanced composite parts are typically in the military and aerospace industries.

As a result of the previously noted issues and due to advances in thermoplastic composites, many product development engineers and design engineers believe that composite materials will play an ever-increasing role in modern technological development. New thermoplastic resins are regularly developed and more innovative methods of manufacturing are being introduced, bringing down the costs of manufacturing parts. As costs for reinforced thermoplastic production are lowered, the use of thermoplastics composites becomes more and more viable in many commercial and industrial applications.

The composites that will be treated and bonded in this project are Carbon/PEEK, Carbon/PPS and Glass/Pa12.

3.1.1 Carbon/Polyether ether Ketone (PEEK)

Datasheet

| Properties | Value |
|----------------------------------------|------------------------|
| Density | 1600 kg/m ³ |
| Young's modulus (<i>E</i>) | 50 GPa |
| Shear strength (<i>longitudinal</i>) | 750 MPa |
| Elongation @ break | 1.8 % |
| Melting point | 340 °C |
| Thermal conductivity @ 23 °C | 0.92 W/m.K |
| Water absorption | 0.14% |

Table 3-1: Datasheet of Carbon/PEEK

Advantages

- Excellent mechanical and chemical resistance
- Retained to high temperatures
- Highly resistant to thermal degradation as well as attack by both organic and aqueous environments
- Chemical and water resistance
- Can be used continuously to 250°C and in hot water or steam without permanent loss in physical properties
- Very low moisture absorption
- Inherently good wear and abrasion resistance

Disadvantages

- Dangerous if it is attacked by halogens as well as some halogenated compounds and aromatic hydrocarbons at high temperatures

Applications

- Bearings
- Piston parts
- Pumps
- Compressor plate valves
- Cable insulation
- Advanced biomaterial used in medical implants
- Extensively used in the aerospace, automotive, electronics and chemical process industries

3.1.2 Carbon/Polyphenylene sulfide (PPS)

Datasheet

| Properties | Value |
|-------------------------------|------------------------|
| Density | 1460 kg/m ³ |
| Modulus (GPa), (longitudinal) | 17.2 GPa |
| Shear strength (longitudinal) | 156 MPa |
| Elongation at break | 1.4 % |
| Melting temperature range | 315-360 °C |
| Thermal Conductivity | 0.850 W/m*K |
| Water absorption | 0.12% |

Table 3-2: Datasheet of Carbon/PPS

Advantages

- Good fracture and impact properties
- Light and strong
- Good weight-to-strength ratio
- Low flex fatigue over time
- It is non flammable
- It is easy to produce in large quantities

Disadvantages

- Expensive

Applications

- Aerospace Industry
- Automobile industry
- Nautical Industry
- Sports Industry
- Military Industry

3.1.3 Glass /Polyamide 12 (Pa12)

Datasheet

| Properties | Value |
|------------------------------------|------------------------|
| Density | 1300 kg/m ³ |
| E- module (E) | 7 GPa |
| Shear strength (σ_t) | 152 MPa |
| Elongation at break | 4.5 % |
| Yield point | 70 MPa |
| Operating temperature (Short term) | 140°C |
| Melting point range | 250-290 °C |
| Thermal Conductivity | 0,40 W/m*K |
| Water absorption | 0.2 % |

Table 3-3: Datasheet of Glass/Pa12

Advantages

- Excellent mechanical and chemical resistance
- Retained to medium temperatures
- As the temperature rises, the polymer's:
 - Elongation increases
 - Creep effects increase
 - Stress relaxation increases
 - Impact strength (toughness) increases
- Skin typically formed because of thermal and viscous boundary layer effects upon injection of molten material into a mold
- Skin consists of highly directional, often fiber-depleted and sometimes crystalline material.
- Colorants are added to give certain desired colors to the polymer.
- Low elongation at break
- High melt viscosity

Disadvantages

- Because of the cross-linked chain structure of polymers, the tensile strength of polymers tends to degrade with increasing temperature.
- As the temperature rises, the polymer's:
 - Modulus (tensile, flexural) values drop
 - Tensile strength drops

- Skin is typically highly influential to the properties of a molded part. This fact makes prototyping (prior to hard tooling) difficult.
- Flame-retardants are added to minimize the chance of a material igniting. For a material to be considered flame-retardant, it needs to meet the UL flammability standards, UL 94.
- Stabilizers are added to inhibit degradation caused due to exposure to Oxygen, sunlight (UV exposure), heat, and water.
- Flowing agents are added to improve flow characteristics during processing.
- Release agents are added to improve mold release characteristics.
- Lubricity agents are added to lower the surface coefficient of friction of the finished product.

Applications

- Seat panel (aerospace)
- Military helmet
- Bicycle helmet
- BMW M3 bumper beam
- Back cover shelf (automotive)
- Nike soccer
- Extensively used in the nautical, aerospace, automotive, military and sport industries.

3.2 Metals

A metal is a chemical element that is a good conductor of both electricity and heat. In a metal, atoms tend to lose electrons to form positive ions. Those ions are surrounded by delocalized electrons, which are responsible for the conductivity.

Metals in general have high electrical conductivity, thermal conductivity, luster and density, and the ability to be deformed under stress without cleaving. The high density of most metals is due to the tightly packed crystal lattice of the metallic structure. There are also several metals that have low density, hardness, and melting points. These metals are called the alkali and alkaline earth metals and are extremely reactive.

A metal alloy is a mixture of two or more elements in solid solution in which the major component is a metal. Most pure metals are either too soft, brittle or chemically reactive for practical use. Combining different ratios of metals as alloys modifies the properties of pure metals to produce desirable characteristics. The purpose of making alloys is generally to make them less brittle, harder, resistant to corrosion, or have a more desirable colour and luster. Of all the metallic alloys in use today, the alloys of iron (e.g. steel, stainless steel and cast iron) make up the largest proportion, both by quantity and commercial value. Iron alloyed with various proportions of carbon gives low, mid and high carbon steels. The addition of silicon will produce cast irons, while the addition of chromium, nickel and molybdenum to carbon steels results in stainless steel.

Other significant metallic alloys are those of aluminum, titanium, copper and magnesium. Copper alloys have been known since prehistory and have many applications today, most importantly in electrical wiring. The other alloys have been developed relatively recently. These alloys are valued for their high strength-to-weight ratios. Magnesium can also provide electromagnetic shielding. These materials are ideal for situations where high strength-to-weight ratio is more important than material cost, such as in aerospace and some automotive applications.

Some metals and metal alloys possess high structural strength per unit mass, making them useful materials for carrying large loads or resisting impact damage. Metal alloys can be engineered to have high resistance to shear, torque and deformation strength. However the same metal can also be vulnerable to fatigue damage through repeated use or from sudden stress failure when a load capacity is exceeded. The strength and resilience of metals has led to their frequent use in high-rise building and bridge construction. The two most commonly used structural metals, iron and aluminum, are also the most abundant metals in the earth's crust.

Metals are, as known, good conductors, making them valuable in electrical appliances and for carrying an electric current over a distance with little energy lost. Electrical power grids rely on metal cables to distribute electricity. Home electrical systems, for the most part, are wired with copper wire for its good conducting properties.

The thermal conductivity of metal is useful for containers to heat materials over a flame. Metal is also used for heat sinks to protect sensitive equipment from overheating.

The high reflectivity of some metals is important in the construction of mirrors, including precision astronomical instruments. This property is also used in the jewelry industry.

The metals that will be treated and bonded with the composites in this project are aluminum 2017 and stainless steel 316L.

3.2.1 Aluminium 2017

Datasheet

| Properties | Values |
|------------------------------|------------------------|
| Density | 2790 kg/m ³ |
| Young's modulus (<i>E</i>) | 72.4 GPa |
| Ultimate Shear Strength | 427 MPa |
| Elongation @ break | 22 % |
| Shear Strength | 262 MPa |
| Specific heat capacity | 0.880 J/g-°C |
| Melting point | 513 - 641 °C |
| Thermal Conductivity | 134 W/m.K |

Table 3-4: Datasheet of Aluminum 2017

Advantages

- Soft, durable, ductile and malleable
- Low density, only one-third of the density and stiffness of steel
- High corrosion resistance, due to a thin surface layer of aluminum oxide that forms when the metal is exposed to air which effectively prevent further oxidation
- Prevent further oxidation due to its normal protection of aluminum oxide
- Good heat conductor
- A nonmagnetic metal
- The most abundant metal and the third most abundant element, after oxygen and silicon
- Non-toxic, used in the packaging industry for food and beverages
- Can appear from silvery to gray depending on the surface roughness

Disadvantages

- One disadvantage of aluminum alloy is the difficulty of making reliable soldered joints. Oxidation of the surface of the heated metal prevents soft solder from adhering to the material

Applications

- Used in structural components in the aerospace industry
- For transportation and buildings
- Automotive industry
- Electrical industry
- An important component of silver paints

3.2.2 Stainless Steel 316L

Datasheet

| Properties | Value |
|------------------------------|------------------------|
| Density | 8000 kg/m ³ |
| Young's modulus (<i>E</i>) | 193 GPa |
| Ultimate Shear Strength | 558 MPa |
| Elongation @ break | 50 % |
| Shear Strength | 186 MPa |
| Specific heat capacity | 500 J/kgK |
| Melting point | 1371- 1399 °C |
| Thermal Conductivity | 16.2 W/m.K @ 100 °C |

Table 3-5: Datasheet of Stainless Steel 316L

Advantages

- High corrosion resistance, formability and ductility
- Contain chromium and nickel as major alloying elements that protect the steel from further oxidation
- Resistance to fire and heat allowing it to resist scaling and retain strength at high temperatures.
- 100 % recyclable
- The austenitic structure make it more resistant to general corrosion and pitting

Disadvantages

- Difficulty in welding due to its fast dissipation of heat which can also produce ruined pieces or high wastage costs
- Difficult to fabricate, it is not as malleable as other metals
- High cost of final polishing and finishing

Application

- Used for jewellers and watches
- Medical and surgical tools and implants
- Architectural applications
- Marine applications
- Food industries

3.3 Surface preparation – Different Treatments

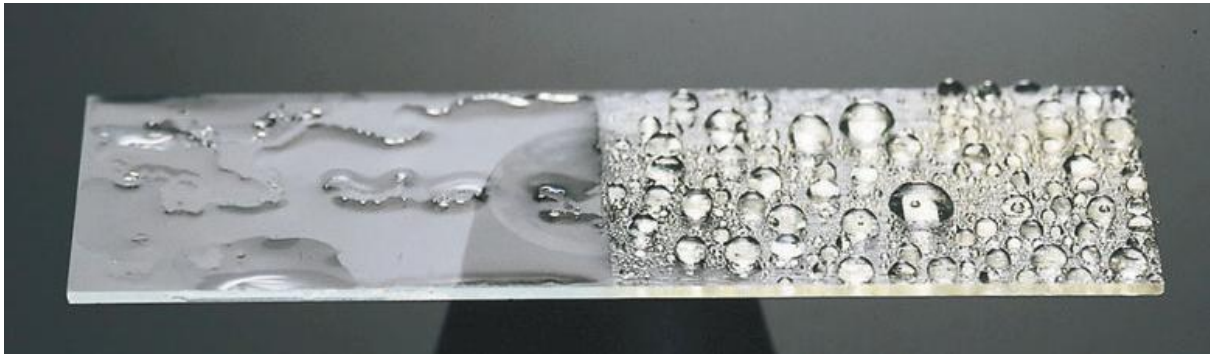


Figure 3-3: Different between a treated (left side) and untreated surface (right side)

Determined bonding processes require special surface preparation, because several materials have a low surface energy. The low surface energy can cause that the adhesive cannot connect with the material's surface so that a stability problem could be a consequence. For this reason the surface properties of the materials are very important for the field of application.

A sufficient surface energy is the basis for a good wettability as well as the stability. With the choose of the right surface treatment it is possible to bond materials which are not at all, or hardly be able to assembled. The interaction between the right surface treatment and the right adhesive leads to an improvement of the stability and a climate stability.

In general you can differ between three different types of surface treatment: chemical, mechanical and physical treatment.

3.3.1 Chemical

This method consists in applying one chemical emulsion to change the surface properties of the metals. First, it is necessary to remove grease and oil solvent. If not enough you must treat the surface with chemical or mechanical means to remove oxides, etc.

This chemical mixture consists in one strong detergent solution that is used to emulsify surface contaminants on both metallic and nonmetallic substrates. These solutions are usually heated. Parts for cleaning are generally immersed in a well agitated solution. The surfaces are then raised immediately with demonized water and dried. Chemical cleaning is often used in combination with other surface treatments, for example abrasion.

Alkaline detergents recommended for prebond cleaning are combinations of alkaline salts such as sodium metasilicate and tetra sodium pyrophosphate with surfactants included. Many commercial detergents are available.

The best method is chemical treatment with chromic mixture. Chromates react with the surface, thus providing protection against oxidation. It is important to stick surfaces immediately after cleaning. The contaminants in the air may reduce adherence to half in less than an hour.

3.3.2 Mechanical

Abrasion

The abrasion is a method that is applied to metal or plastic surfaces to modify the surface characteristics. Superficial scraping of metal and plastic surfaces using an abrasive, improves the performance in the joint by glue. Sometimes, the abrasion is used to match an irregular or rough surface to remove corrosion. However, the use of this method is inappropriate for materials less than 2 mm thick. This procedure is easily paced in production and it is good for the environment.

The first objective of the abrasion method is to eliminate the impurities and create a homogenous surface.



Figure 3-4: Sandpaper for manual abrasion

Sandblasting

Abrasive blasting is the operation of forcibly propelling a stream of abrasive material against a surface under high pressure to smooth a rough surface, roughen a smooth surface, shape a surface, or remove surface contaminants. There are different types of abrasives:

Mineral: Silica sand is the most commonly used type of mineral abrasive. It tends to break up quickly, creating large quantities of dust, exposing the operator to the potential development of silicosis, a debilitating lung disease. To counter this hazard, silica sand for blasting is often coated with resins to control the dust.

Another common mineral abrasive is garnet. Garnet is more expensive than silica sand, but if used correctly, will offer equivalent production rates while producing less dust and no safety hazards from ingesting the dust. Magnesium sulphate (kieserite) is often used as an alternative to baking soda.

Agricultural: Typically, crushed nut shells or fruit kernels. These soft abrasives are used to avoid damaging the underlying material such when cleaning brick or stone, removing graffiti, or the removal of coatings from printed circuit boards being repaired.

Synthetic: This category includes corn/wheat starch, sodium bicarbonate and dry ice. These "soft" abrasives are also used to avoid damaging the underlying material such when cleaning brick or stone, removing graffiti, or the removal of coatings from printed circuit boards being repaired.

Additional synthetic abrasives include process byproducts (e.g. copper slag, nickel slag and coal slag), engineered abrasives (e.g. silicon carbide, aluminum oxide, glass beads, ceramic shot/grit) and recycled products (e.g. plastic abrasive, glass grit).

Metallic: Steel shot, steel grit, stainless steel shot, cut wire, copper shot, aluminum shot, zinc shot.

Many coarser media used in sandblasting often result in energy being given off as sparks or light on impact. The colours and size of the spark or glow varies significantly, with heavy bright orange sparks from steel shot blasting, to a faint blue glow (often invisible in sunlight or brightly lit work areas) from garnet abrasive.

3.3.3 *Physical*

Thermally and electrical treatment methods are very important nowadays. They are applied in all application areas requiring high strength, reproducibility, durability and climatic resistance.

Flame

Flame treatment was initially developed in the 1950s to improve the surface adhesion properties of polyolefin films. It is a method by chemically changing the surface molecular structure of a substrate in a controlled manner to increase the surface energy and the wettability.

The treatment can be applied to any plastic or metallic component, like bumper fascias, body side moldings, wheel covers, air spoilers, air scoops, door handles, door panels, consoles, dashboards, post trims and carpet backing.

The treatment has many benefits, it has low capital cost, is environmentally friendly, reaches deep recesses, no hazardous byproducts and it is a rapid treatment.

For the flame treatment you need a burner with an open and proper burning flame. When the flame touches the surface of the material, chemical polar groups arise. Hereby the wetting and adhesion properties getting better and the surface energy increase too.

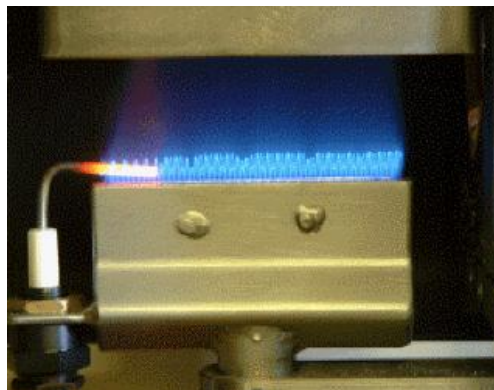


Figure 3-5: Flame machine

Corona

A corona treating system is designed to increase the surface energy of e.g. plastic films, foils and paper in order to allow improved wettability and adhesion of inks, coatings and adhesives.

It is a high frequency electric discharge towards a surface. The result from this is an improvement of the chemical connection between the molecules in the plastic and the applied media/liquid.

This surface treatment neither reduces nor changes the strength and appearance of the material.

During the corona treatment, electrons are accelerated into the surface of the plastic, causing the long chains in the material to rupture, which produce a multiplicity of open valences. The ozone from the electrical discharge creates an oxygenation, which in turn forms new carbonyl groups with a higher surface energy.

A corona treating system consists of two major components: the power supply and the treater station. The power supply accepts standard 50/60 Hz utility electrical power and converts it into single phase, higher frequency power that is supplied to the treater station. The treater station applies this power to the surface of the material, through an air gap, via a pair of electrodes at high potential.



Figure 3-6: Example of a Corona machine

Plasma

By adding energy the states changes from solid to liquid and from liquid to gas. The fourth state, the plasma state, is created by adding further energy to the gas that then becomes ionized. Plasma is a substance where many of the atoms or molecules are effectively ionized, allowing charges to flow freely. This collection of charged particles containing about equal numbers of positive ions and electrons exhibits. With the addition of heat, a significant number of atoms release some or all of their electrons. This leaves the remaining parts of those atoms with a positive charge, and the detached negative electrons are free to move. These atoms and the resulting electrically charged gas are said to be ionized. The gas becomes plasma when enough atoms are ionized to a point that significantly affects the electrical characteristics of the gas. Plasmas can carry electrical currents and generate magnetic fields and the most common method for producing plasma is by applying an electric field to a gas in order to accelerate the free electrons.

By using the plasma treatment technology on material surfaces, the added energy from the plasma state can be transferred to these surfaces through a generated electrically charged atmosphere, similar to corona treatment. This process increases the surface energy of materials.

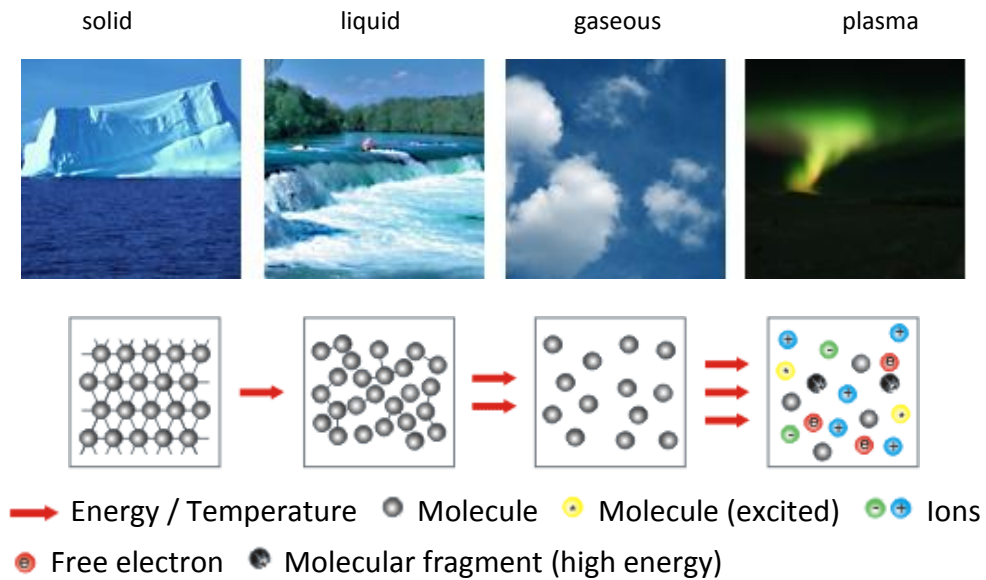


Figure 3-7: The four different states

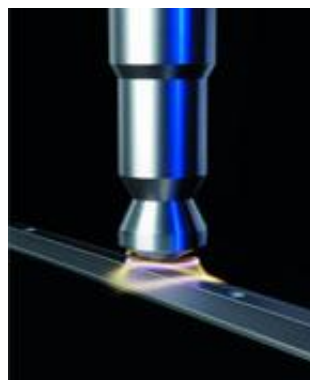


Figure 3-8: Plasma machine

Laser

Usually, laser irradiation is used for cleaning, structuring, chemical and morphological modification and curing of material surfaces. As a treatment for adhesive bonding, laser radiation generates an oxide and grease free surface with a possible micro-structuring. A wet chemical cleaning is unnecessary. Laser ablation works noise and touch free with small running cost. Precise control makes the integration in serial productions possible.

The functional principle of removing coating layers is the followings: The coating layer is removed by vaporization through absorbing the laser spot. The blank base material reflects the laser radiation, so that the ablation process stops automatically. Very short laser pulses cause a very little thermal influence on the base material. It is possible to treat the metal without damaging or melting of the base material.

Laser radiation offers the possibility to treat the material surfaces selectively with a high flexibility in order to improve the adhesion characteristics. This has the advantage in comparison to other procedures that only the required surfaces need to be processed.



Figure 3-9: Laser treatment machine

3.4 Surface Energy

The surface energy depends of the attraction between the solid and the liquid. If the molecules of the liquid have a stronger attraction to the molecules of the solid surface than to each other (the adhesive forces are stronger than the cohesive forces), wetting of the surface occurs. Alternately, if the liquid molecules are more strongly attracted to each other than the molecules of the solid surface (the cohesive forces are stronger than the adhesive forces), the liquid beads-up and does not wet the surface of the part.

One way to quantify a liquid's surface wetting characteristics is to measure the contact angle of a drop of liquid placed on the surface of an object. The known properties of the liquids and the measured contact angles are used to calculate surface free energy. The contact angle is the angle formed by the solid/liquid interface and the liquid/vapor interface measured from the side of the liquid, see figure 3-10. For a material to be effective, the contact angle should be as small as possible.

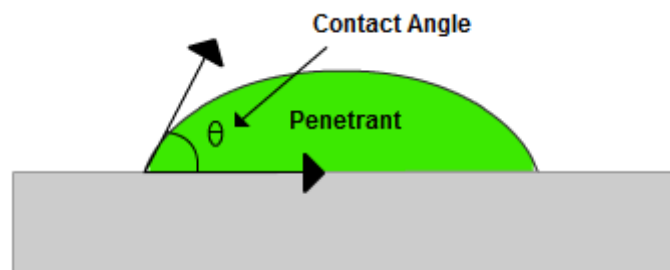


Figure 3-10: Measuring the contact angle

When the liquid encounters another substance, there is usually an attraction between the two materials. The adhesive forces between the liquid and the second substance will compete against the cohesive forces of the liquid. Liquids with weak cohesive bonds and a strong attraction to another material will tend to spread over the material. Liquids with strong cohesive bonds and weaker adhesive forces will tend to bead-up or form a droplet when in contact with another material.

3.4.1 Drop test

A way to calculate the surface energy is to do drop tests and to calculate the contact angle. In this project the machine DIGIDROP – Contact Angle Meter, with related software, Digidrop, was used for doing the drop tests.



Figure 3-11: Drop test machine

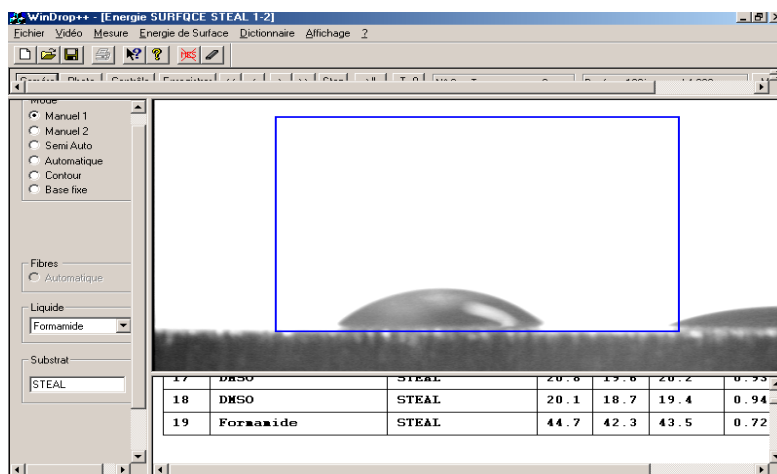


Figure 3-12: The software Digidrop

Multiple analysis methods exist to measure the surface energy, making the choice of method and analysis important. The methods that are used in this project are Owens-Wendt for the composites and Fowkes method for the metals. When calculating the total energy of a surface the total energy is the sum of the polar composite and the dispersive composite.

Owens-Wendt

The Owens-Wendt approach is one of the most common methods for calculating the surface free energy of polymeric materials. The most frequently measuring liquids that being used is water and diiodomethane. Through the Owens-Wendt model, the total surface energy can be expressed as the sum of contributions from dispersive and polar force components as

$$\gamma_s = \gamma_s^d + \gamma_s^{nd}$$

γ_s^d — Dispersive component

γ_s^p — Non dispersive component

The equation between tension component and contact angle is

$$\gamma_L(1 + \cos\theta) = 2\sqrt{\gamma_S^d}\sqrt{\gamma_L^d} + 2\sqrt{\gamma_S^p}\sqrt{\gamma_L^p}$$

The model requires the measure of contact angle from at least two different liquids to calculate the surface energy.

Fowkes method

The Fowkes principal equation is mathematically equivalent to Owens-Wendt but it is still a different method to calculate the surface energy since it is derived from different principles than Owens-Wendt. The different to Owen-Wendt method is that it is also possible to simply do tests using liquids with no polar component to their surface energies, and then liquids that have both polar and dispersive components.

3.5 Adhesives

Adhesives were first used many thousands years ago, and most were derived from naturally occurring vegetable, animal, or mineral substances. Synthetic polymeric adhesives displaced many of these early products due to stronger adhesion and greater resistance to operating environments.

An adhesive, or glue, is a mixture in a liquid or semi-liquid state that adheres or bonds items together. The types of materials that can be bonded are vast but they are especially useful for bonding thin materials. Adhesives can cure (harden) for example by evaporating a solvent or by chemical reactions that occur between two or more constituents.

Adhesives are an advantageous for joining thin or dissimilar materials, minimizing weight, and when a vibration dampening joint is needed. A disadvantage to adhesives is that they do not form an instantaneous joint, unlike most other joining processes, because the adhesive needs time to cure.

The successful application of an adhesive depends on many factors. Anyone using an adhesive faces a complex task of selecting the proper adhesive and the correct processing conditions that allow a bond to form. One must also determine which substrate-surface treatment will permit an acceptable degree of permanence and bond strength.

The adhesive joint must be correctly designed to avoid stresses within the joint that could cause premature failure. Also, the physical and chemical stability of the bond must be forecast with relation to its service environment.

Advantages and disadvantages of Adhesive Bonding – Why bonding?

| Advantages | Disadvantages |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <ul style="list-style-type: none"> • Provides large stress-bearing area • Provides excellent fatigue strength • Damps vibration and absorbs shock • Joins all shapes and thicknesses • Provides smooth contours • Seals joint • Joins any combination of similar and dissimilar materials • Often less expensive and faster than mechanical fastening • Provides attractive strength-to-weight ratio | <ul style="list-style-type: none"> • Surfaces must be carefully cleaned • Long cure times may be needed • Limitation on upper continuous operating temperature • Heat and pressure may be required • Jigs and fixtures may be needed • Rigid process control usually necessary • Inspection of finished joint is difficult • Useful life depends on environment • Environmental, health, and safety considerations are necessary |

Table 3-6: Advantages and disadvantages of adhesive bonding

3.5.1 Theories of adhesion

The mechanism of adhesion has been investigated for years; several theories have been proposed in an attempt to provide an explanation for adhesion phenomena. However, no single theory explains adhesion in a general, comprehensive way.

The bonding of an adhesive to an object or a surface is the sum of a number of mechanical, physical, and chemical forces that overlap and influence one another. As it is not possible to separate these forces from one another, one distinguishes between mechanical interlocking, caused by the mechanical anchoring of the adhesive in the pores and the uneven parts of the surface; electrostatic forces, as regard to the difference in electro negativities of adhering materials and the other adhesion mechanisms dealing with intermolecular and chemical bonding forces that occur at the interfaces of heterogeneous systems.

This chemical adhesion mechanism is explained in the case of the intermolecular forces by the adsorption theory and in the case of chemical interactions by the chemisorption theory. The processes that play a role in the bonding of similar types of thermoplastic high-polymer materials, e.g. homogeneous systems, can be determined with the diffusion theory.

Mechanical interlocking

The mechanical interlocking theory of adhesion states that good adhesion occurs only when an adhesive penetrates into the pores, holes and crevices and other irregularities of the adhered surface of a substrate, and locks mechanically to the substrate. The adhesive must not only wet the substrate, but also have the right rheological properties to penetrate pores and openings in a reasonable time.

This theory explains a few examples adhesion such as rubber bonding to textiles and paper. Since good adhesion can occur between smooth adherend surfaces as well, it is clear that while interlocking helps promote adhesion, it is not really a generally applicable adhesion mechanism.

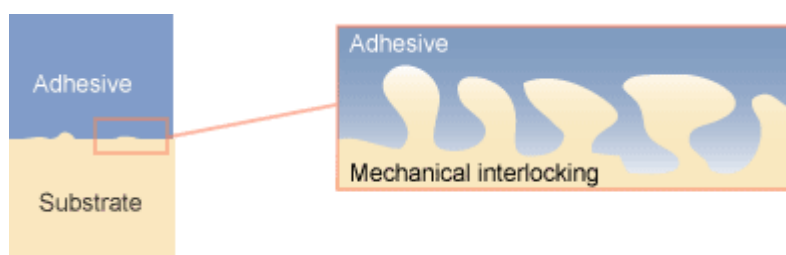


Figure 3-13: Surface of a material

Treatment methods applied on surfaces enhance adhesion. These treatments (especially plastic surface treatments) result in micro roughness on the adherend surface, which can improve bond strength and durability by providing mechanical interlocking. Beyond mechanical interlocking, the enhancement of the adhesive joint strength due to the roughing of the adherend surface may also result from other factors, such as formation of a larger surface, improved kinetics of wetting and increased plastic deformation of the adhesive.

Adsorption

The adsorption theory states that adhesion results from intimate intermolecular contact between two materials, and involves surface forces that develop between the atoms in the two surfaces.

This theory is the most important mechanism in achieving adhesion. The most common surface forces that form at the adhesive-adherend interface are van der Waals forces. In addition, acid-base interactions and hydrogen bonds, generally considered a type of acid-base interaction, may also contribute to intrinsic adhesion forces. Research has experimentally demonstrated that the mechanism of adhesion in many adhesive joints only involves interfacial secondary forces. The calculated attractive forces between two surfaces are considerably higher than the experimentally measured strength of adhesive joints. This discrepancy between theoretical and experimental strength values has been attributed to voids, defects or other geometric irregularities which may cause stress concentrations during loading.

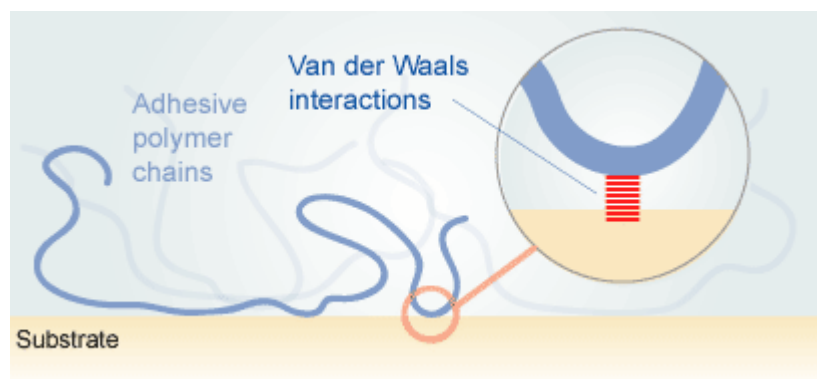


Figure 3-14: Van der Waals interactions

To obtain good adsorption, intimate contact must be reached such that van der Waals interaction or the acid-base interaction or both take place; hence good wetting is essential. According to Young's equation, the surface tensions (liquid/gas: γ_{LG} , solid/liquid: γ_{SL} and solid/gas: γ_{SG}) at the three phase contacts are related to the equilibrium contact angle through:

$$\gamma_{SG} = \gamma_{SL} + \gamma_{LG} \cdot \cos\theta$$

One important factor that influences the adhesive joint strength is the ability of the adhesive to spread spontaneously on the substrate when the joint is initially formed. For spontaneous wetting to occur:

$$\gamma_{SG} \geq \gamma_{SL} + \gamma_{LG}$$

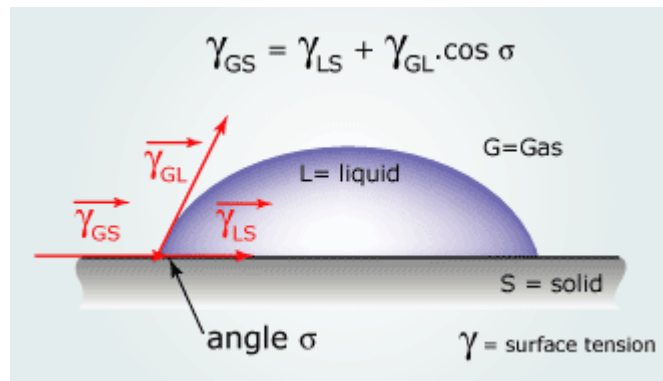


Figure 3-15: Angle of contact of a drop of liquid with the surface of a solid object

One can say that for a good wetting γ_{SG} must be bigger than γ_{LG} ($\gamma_{SG} > \gamma_{LG}$).

Generally speaking, the liquid surface tension of the adhesive should be less than the critical wetting tension of the solid surface of the substrate.

Electrostatic

The basis of the electrostatic theory of adhesion is the difference in electro negativities of adhering materials. Adhesive force is attributed to the transfer of electrons across the interface, creating positive and negative charges that attract one another. For example, when an organic polymer is brought into contact with metal, electrons are transferred from metal into the polymer, creating an attracting electrical double layer (EDL).

The electrostatic theory tell us that these electrostatic forces at the interface (i.e. in the EDL), account for resistance to separation of the adhesive and the substrate.

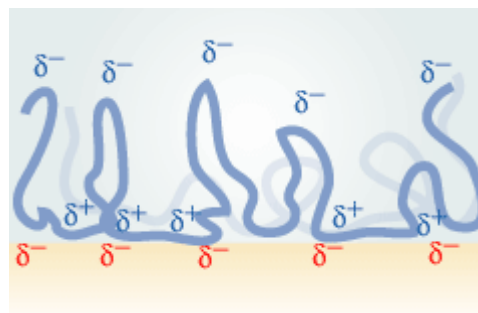


Figure 3-16: Electrostatic theory

Some controversies have arisen surrounding the electrostatic theory owing to the fact that the EDL could not be identified without separating the adhesive bond. Additionally the effect of the electrical double layer on the adhesive bond strength was exaggerated, as argued by many researchers.

Diffusion

The fundamental concept of the diffusion theory is that adhesion arises through the interdiffusion of molecules in the adhesive and adherend. The diffusion theory is primarily applicable when both the adhesive and adherend are polymeric, having long-chain molecules capable of movement. Bonds formed by solvent or heat welding thermoplastic result from the diffusion of molecules.

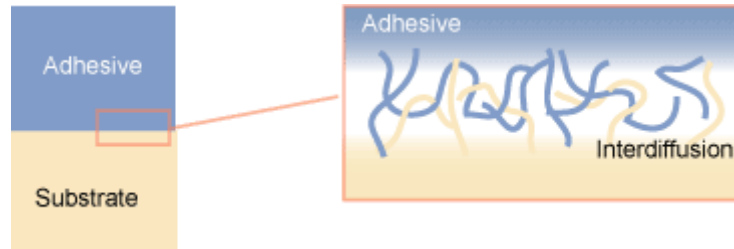


Figure 3-17: Interdiffusion of the substrate and the adhesive

Parameters affecting the diffusion process are: contact time, temperature, molecular weight of polymers and physical form (liquid, solid). Polarity generally increases adhesion.

Chemisorption

The chemical bonding mechanism suggests that primary chemical bonds may form across the interface. Chemical bonds are strong and make a significant contribution to the intrinsic adhesion in some cases.

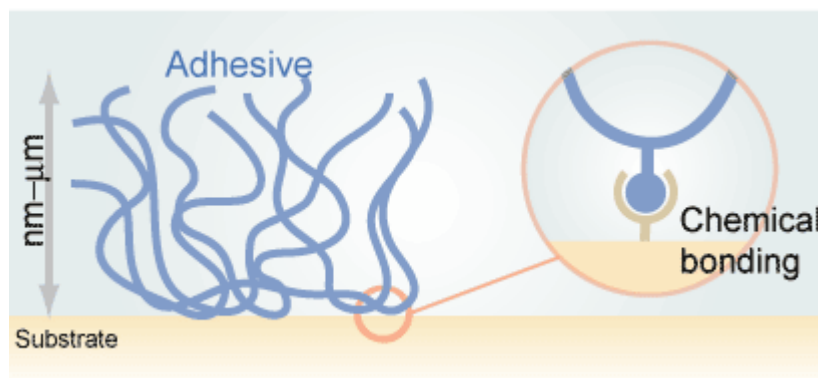


Figure 3-18: Chemical bonding

For example, primary chemical forces have energies ranging between 60-1100 kJ/mol, which are considerably higher than the bond energies secondary forces (0.08-5 kJ/mol). One should also mention the coupling agents and adhesion promoters that are used to help in fixing the adhesive at the surface by chemical reaction.

3.5.2 Adhesive Selection

Factors most likely to influence adhesive selection are the stress, chemical factors, exposure, temperature, biological factors and working properties. Regarding to the controlling factors involved, the many adhesive available can usually be narrowed to a few candidates that are most likely to be successful. The general areas when selecting adhesives should be the material to be bonded, service requirements, production requirements, and overall cost.

Adhesive Classification

Adhesive may be classified by many methods. The most common methods are by function, chemical composition, mode of application and setting, and end use.

The functional classification defines adhesives as being structural or non-structural. Structural adhesives are materials of high strength and permanence. Their primary function is to hold structures together and be capable of resisting high loads. Non-structural adhesives are not required to support substantial loads. They merely hold light-weight materials in place or provide a seal without having a high degree of strength.

The chemical composition classification broadly describes adhesives as thermosetting, thermoplastic, elastomeric, or combinations of these. There are then many chemical types within each classification. A thermoplastic becomes liquid when it reaches higher temperatures whereas a thermosetting adhesive becomes hard with higher temperatures. The thermosetting is hence used for high temperature applications and also for high stress. The elastomeric however is a polymer with the property of viscoelasticity. The primary use is so for seals, adhesives, and molded flexible parts.

In the following part we will have a look at the different groups of thermosetting adhesives. Only this group will be interesting for the project because it supports the high temperature use and also the required materials plastics and metals. The thermoplastic adhesives are not suitable in the most cases because of a low temperature resistance or because they do not bond metal and plastic in the same time.

In the following table 3.7, you can see the groups of thermosetting adhesives with a description, special characteristics and the usual materials.

| Adhesive | Description | Special Characteristics | Usual materials |
|-----------------------------------------------|----------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------|
| Cyanoacrylate | One-part liquid | Fast setting, good bond strength; poor heat and shock resistance | Metals, plastics, glass |
| Polyester | Two-part liquid or paste | Resistance to chemicals, moisture, heat, weathering; wide range of strength; some resins do not fully cure in presence of air | Metals, foils, plastics, plastic laminates, glass |
| Urea Formaldehyde | Usually supplied as two-part resin and hardening agent | Not as durable as others but suitable for fair range of service conditions | Plywood |
| Melamine formaldehyde | Powder to be mixed with hardening agent | Equivalent in durability and water resistance to phenolics and resorcinols | Plywood, other wood products |
| Resorcinol and phenol-resorcinol formaldehyde | Usually alcohol-water solutions to which formaldehyde must be added | Suitable for exterior use, bond strength equals or betters strength of wood, do not bond directly to metal | Wood, plastics, fibreboard, plywood |
| Epoxy | Two-part liquid or paste; one-part liquid, paste or solid; solutions | Most versatile adhesive available; excellent tensile shear strength ; poor peel strength, excellent resistance to moisture and solvents, low cure shrinkage, variety of curing agents/hardeners results in many variations | Metals, plastic, glass, rubber, wood, ceramics |
| Polyimide | Support film, solvent solution | Excellent thermal and oxidation resistance, suitable for continuous use at 310°C and short-term use to 660°C; very expensive | Metals, metal foil, honeycomb core |
| Polybenzimidazole | Supported film | Good strength at high temperatures, suitable for continuous use at 210°C and short term 760°C; expensive | Metals, metal foil, honeycomb core |
| Acrylic | Two-part liquid or paste | Excellent bond to many plastics, good weather resistance, fast cure, poor peel and impact strength | Metals, many plastics, wood |
| Acrylic acid diester | One-part liquid or paste | Chemically blocked, anaerobic type, excellent wetting ability, useful temperature range of -300°C to +60°C; withstands rapid thermal cycling, high-tensile-strength grade requires cure at 10°C, cures in minutes at 40°C, very expensive | Metals, plastics, glass, wood |

Table 3-7: Groups of thermosetting adhesives

3.6 Possible bond failures

Basic fracture modes are shown in figure 3-19. They may be classified according to NF EN ISO 10365 and will bear the following abbreviations:

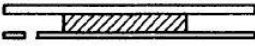
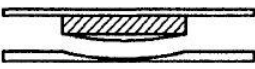
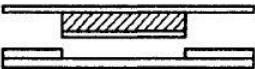
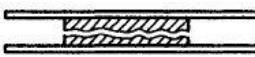

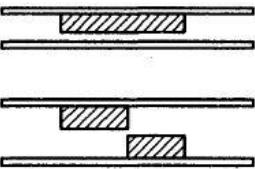
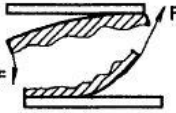
| | Faciès de rupture | Désignation |
|----------|-----------------------------------------------------------------------------------------------------------------------------------------------|-------------|
| Substrat |  Rupture de l'un ou des deux supports | SF |
| |  Rupture de cohésion du support | CSF |
| |  Rupture par délamination | DF |
| Adhésif | <p>Types de rupture de cohésion</p>  Rupture de cohésion | CF |
| |  Rupture de cohésion spéciale | SCF |
| |  Rupture d'adhésion | AF |
| |  Rupture de cohésion avec pelage | ACFP |

Figure 3-19: Possible bond failure

- Substrate failure (sf): under some circumstances the adhesive itself and the interface are strong enough to sustain the applied load. In this case the stress concentration at the end of overlap causes fracture within the substrate (shear bar).
- Delamination failure(df): the delamination failure is when a part of the adherent getting ripped by the other part
- Cohesive failure (cf): in this case fracture propagates within the layer of adhesive so that both halves of shear bar show a layer of adhesive.
- adhesive failure (af): here the adhesion of the adhesive to the substrate at the interface is the weak link. Upon rupture the entire layer of adhesive sticks to the shear bar. There is no crack propagation within the adhesive.
- Adhesive/cohesive failure (acf): this is a mixed mode failure where fracture propagation jumps back and forth between fracture at the interface and fracture within the adhesive.

Since each test was repeated several times it may well be, that different samples showed more than one fracture mode. Sometimes one sample can even include more failure modes like in figure 3.20 showed. Then it will be written like af50% + cf50% to explain that 50% of the failure is an adhesive failure and 50% is a cohesive failure.

If different materials are tested the metal will have an 'a' and the composite a 'b'. For example afb100% says that there is a 100% adhesive failure on the composite side.

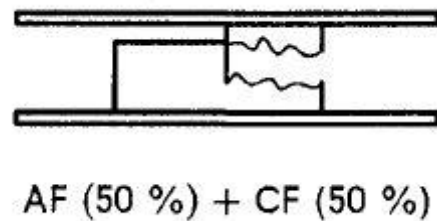


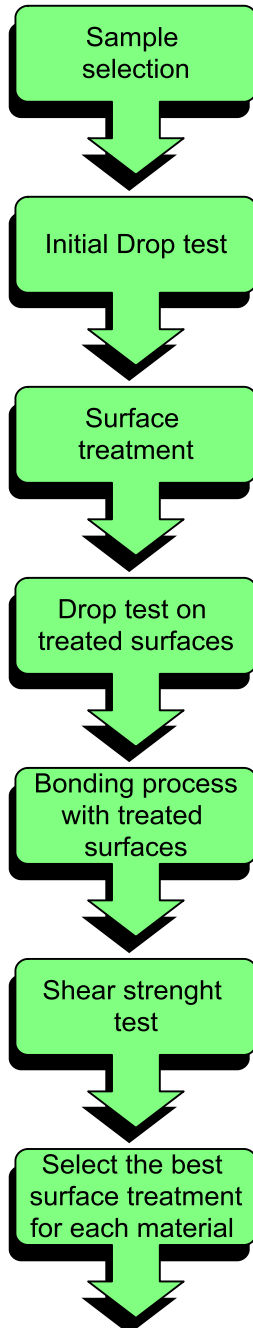
Figure 3-20: Combination of failure modes

Adhesive joints may fail adhesively or cohesively. Adhesive failure (af) is interfacial bond failure between the adhesive and the material (adherend). Cohesive failure (cf) occurs when the adhesive fractures, allowing a layer of adhesive to remain on both substrates. When the adherend fails before the adhesive, it is known as a substrate failure (sf).

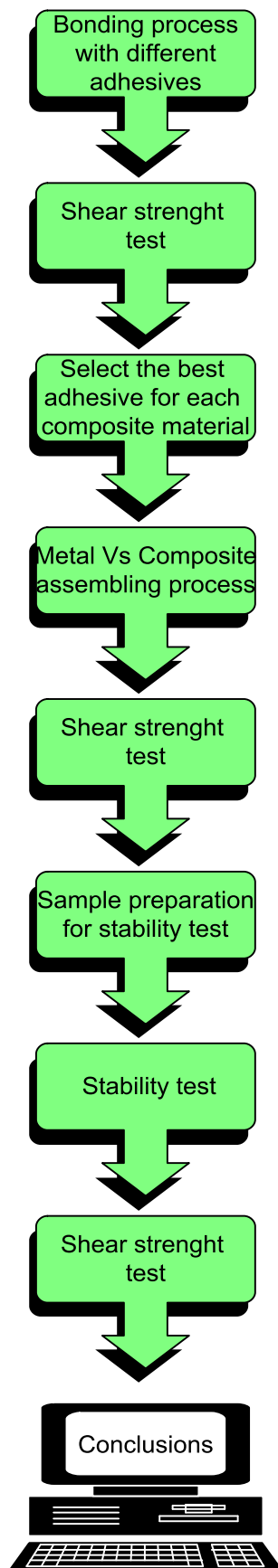
Cohesive failure within the adhesive or one of the adherend is the ideal type of failure, because the maximum strength of the materials in the joint has been reached. However, failure mode should not only be used as a criterion for a useful joint. Some adhesive-adherend combinations may fail in adhesion but provide sufficient strength margin to be practical. But anyway an analysis of failure mode can be an extremely useful tool to determine if a failure is due to a weak boundary layer or improper surface preparation. It is hard to determine the exact cause of adhesive failure, because so many factors in adhesive bonding are interrelated.

4 PROCESS DESCRIPTION

In this paragraph all the technical process steps are defined:



- The first part is to select the kind of samples that we are going to use, the size of them and the amount that we will need for the whole processes during the project. You can see the total sample amount in table 4.1.
- Next step is to make the initial drop test on each material surface to have an initial data of the current surface energy.
- After the research studies we will treat each material with all the available surface treatments methods.
- To compare with the initial drop test results we will test the surface energy on the treated surfaces. The best treatments will be bonded and tested with shear strength test.
- To check if the drop test results are the right ones or not, we will bond the materials among themselves (i.g. aluminum with aluminum) after applying the best surface treatment on each of them.
- The shear strength test will give us the results of the assembling process.
- The bonding process with the best strength test results is going to be the ones with the best surface treatment.



- Once we have the best surface treatment defined we will continue the process applying this surface treatment to the materials and bond them with different adhesives.
- After the bonding process with the different adhesives we will test them through shear strength test.
- Comparing the results that we will obtain with the shear strength test we will be able to define the best adhesive.
- Once we have the best surface treatment and the best adhesive for each material we will go ahead with the main assembling process, which means to bond the composite with the metal.
- This test will give us the final results. In this step we will see if we are successful on our technical requirements or not.
- We will prepare samples to check the stability of the bonding process in stress conditions. It means surface treatment and bonding process.
- The samples will be put in different chambers to make stability tests.
- The shear strength test after the stability test will give us the information if the samples are resistant or not due to the stress conditions.
- A final matrix with all the results will be fulfilled and presented.

Total sample amount

| | PEEK | PPS | Pa12 | Aluminium | Stainless Steel |
|----------------------------|-----------|-----------|-----------|------------|-----------------|
| Drop test | 11 | 11 | 11 | 3 | 3 |
| Shear strength test | 48 | 48 | 48 | 117 | 27 |
| Stability test | 24 | 24 | 24 | 36 | 36 |
| TOTAL | 83 | 83 | 83 | 156 | 66 |

Table 4-1: Table of total sample amount

4.1 Finding best surface treatment

The first step to find the best bonding process is to find out the best surface treatment. This will be done by doing the drop test and after this, by doing the shear strength test.

The treatments that were made were corona, abrasion, flame, plasma, sandblasting and chemical for the different materials. Before the treatment all the samples were cleaned with Isopropanol to remove any grease or pollution.

4.1.1 Treatment description

- Corona – The corona treatment was used for the composites where we passed the samples with the corona machine three times with a distance of approximately 3-4 cm.
- Abrasion – For the abrasion treatment we followed the standard “NF EN 13887 January 2004” for all the materials. The grain size should be between 45 micro meters to 106 micrometers. For the composites it was used the Norton 100 (μm) and for the metals, Emery Cloth, Grit 100 (μm) and KWB, blatt Schleifstreifen 80 (μm). The materials were cleaned before and after the treatment with Isopropanol. The cleaning before the abrasion is important to get off all the pollutions to avoid that it will be pressed in the materials surface.
- Flame – During the flame treatment we used a fixed burner. The composites were passed three times for each sample with a distance of approximately 2 cm while touching the blue flame.
- Plasma – For the plasma treatment we rented a plasma machine from the plasmatrete company (www.plasmatrete.com) from Paris. In the beginning it was only planned to do the

plasma treatment for the composites but we also did it for the metals after the contact and suggestions from our supplier. We tried different speeds and distances when we passed the materials through the plasma and found out that the best solution was with a speed of 6 m/min with a distance of 1 cm from the surface.

- Sandblasting – We sent in samples of the composites to a sandblasting company through Technacol that made the treatment for us.
- Chemical – For the chemical treatment we followed the standard “NF EN 13887 January 2004” for the metals.
- Laser – We also wanted to try laser treatment on the surfaces and found one supplier and tried to get contact with this supplier but without any success.

4.1.2 Drop test results

All the drop tests were made following the European Standard, NF EN 828. The standard includes calculations from the Zisman method but we are using the Fowkes and the Owens-Wendt method for the composites and the metals. The drop test includes four different standard liquids and three drops of each liquid.

The liquids are:

- Glycerol
- Formamide
- Diiodomethane
- Dimethylsulfoxide

The drop tests were made immediately after the surface treatments to ensure to not lose the changed surface properties. The surface of the material was cleaned before the drop test with Isopropanol and paper to remove any grease and pollution that may affect the surface and the drop test results.

There are different methods to measure the surface energy, Owens-Wendt method which is often used for composite materials and the Fowkes method for the metals.

The following pages show the summary of all the results for the drop tests, including the initial drop test without treatment and drop test after different treatment for all of our materials. For the exact values from the tests see each material appendix.

See appendix 3 for exact values from all the tests.

| Drop test | Date (2010) |
|--------------------|-------------|
| without treatment | Nov 15 |
| after corona | Nov 15 |
| after abrasion | Nov 17 |
| after flame | Nov 26 |
| after plasma | Dec 09 |
| after sandblasting | Dec 09 |

Table 4-2: Dates when the drop tests were made for Carbon/PEEK

| Treatment | Owen-Wendt [mJ/m^2] | |
|-------------------|---------------------------------------|-----------------|
| | Total energy | Polar component |
| Without treatment | 46,466 | 0,4522 |
| Corona | 56,939 | 10,4332 |
| Abrasion | 51,831 | 2,8012 |
| Flame | 62,703 | 15,9825 |
| Plasma | 58,318 | 12,13 |
| Sandblasting | 52,6806 | 2,8378 |

Table 4-3: Sum of the Owen-Wendt results of Carbon/PEEK

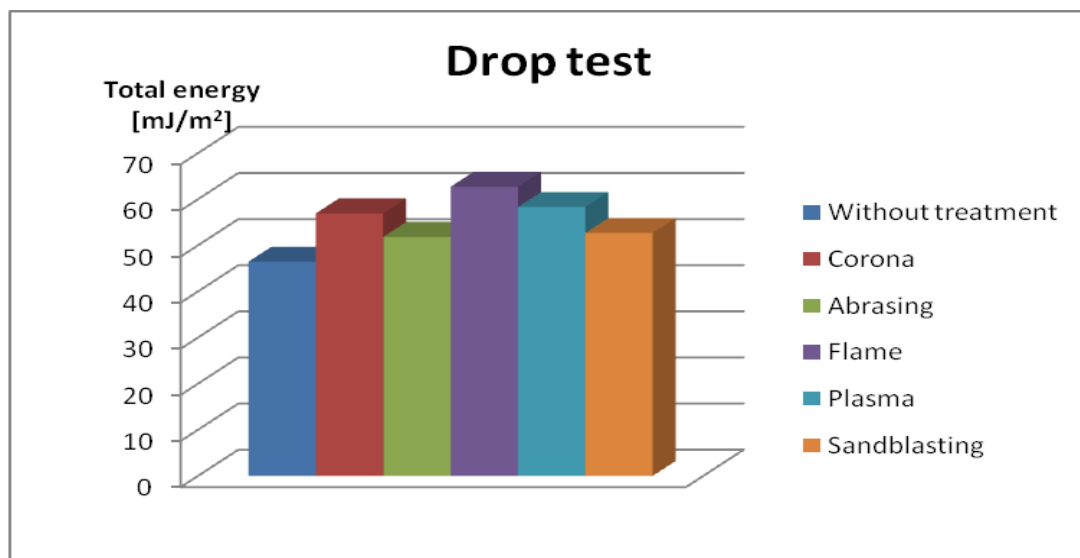


Figure 4-1: Diagram of the total energy by Owen-Wendt method of Carbon/PEEK

The given table and diagram above, shows that the surface energy increases with approximately 12 units after the plasma treatment and with 16 units after the flame treatment regarding to the initial test. The worst surface treatment is the abrasion and the sandblasting, which means that these treatments will be excluded for the shear strength test. The corona and plasma treatments have almost the same results but the plasma is still a little better than corona. Therefore the treatments

that will be used for the shear strength test are the flame and the plasma treatments, to find out which of these two is the best one.

Carbon/PPS

See appendix 4 for exact values from all the tests.

| Drop test | Date |
|--------------------|--------|
| without treatment | Nov 15 |
| after corona | Nov 15 |
| after abrasion | Nov 17 |
| after flame | Nov 26 |
| after plasma | Dec 09 |
| after sandblasting | Dec 09 |

Table 4-4: Dates when the drop tests were made for Carbon/PPS

| Treatment | Owen-Wendt [mJ/m^2] | |
|-------------------|---------------------------------------|-----------------|
| | Total energy | Polar component |
| Without treatment | 45,5318 | 0,5407 |
| Corona | 54,1527 | 8,1158 |
| Abrasion | 50,0997 | 0,6389 |
| Flame | 60,142 | 14,5463 |
| Plasma | 58,9667 | 12,4097 |
| Sandblasting | 50,8327 | 0,4853 |

Table 4-5: Sum of the Owen-Wendt results of Carbon/PPS

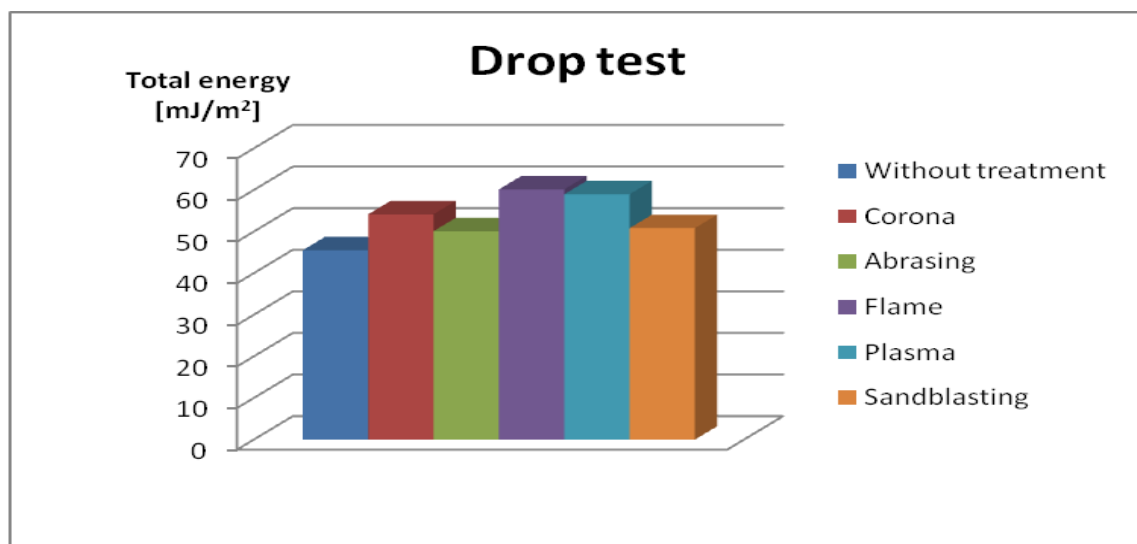


Figure 4-2: Diagram of the total energy by Owen-Wendt method of Carbon/PPS

The flame and the plasma treatment are once again the best ones and will be tested in the shear strength test.

See appendix 5 for exact values from all the tests.

| Drop test | Date |
|--------------------|--------|
| without treatment | Nov 15 |
| after corona | Nov 15 |
| after abrasion | Nov 17 |
| after flame | Nov 26 |
| after plasma | Dec 09 |
| after sandblasting | Dec 09 |

Table 4-6: Dates when the drop tests were made for Glass/Pa12

| Treatment | Owen-Wendt [mJ/m^2] | |
|-------------------|---------------------------------------|-----------------|
| | Total energy | Polar component |
| Without treatment | 41,3159 | 1,51163 |
| Corona | 46,0764 | 2,23594 |
| Abrasion | 45,6636 | 0,79074 |
| Flame | 54,2755 | 9,48872 |
| Plasma | 59,6326 | 12,659 |
| Sandblasting | 51,3249 | 3,3648 |

Table 4-7: Sum of the Owen-Wendt results of Glass/Pa12

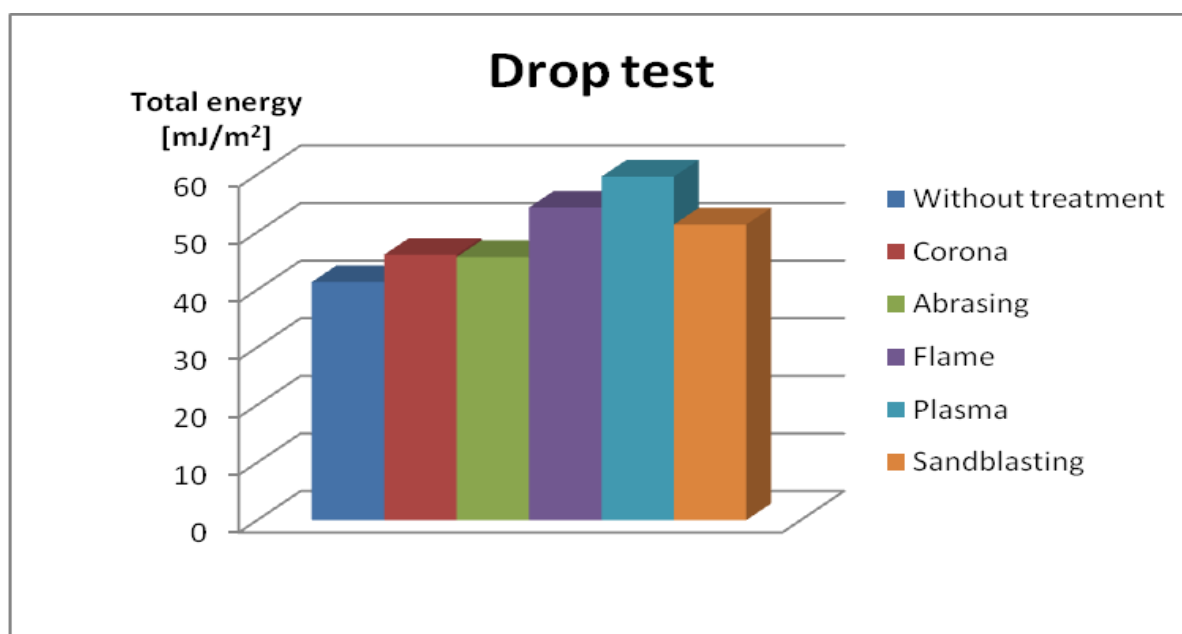


Figure 4-3: Diagram of the total energy by Owen-Wendt method of Glass/Pa12

For the Pa12 composite, the plasma and the flame treatment is superior to the others regarding the total energy and will therefore be selected for the shear strength tests.

Aluminium 2017

See appendix 6 for exact values from all the tests.

| Drop test | Date (2010) |
|-------------------|-------------|
| without treatment | Nov 15 |
| Abrasion (80) | Nov 17 |
| Abrasion (100) | Nov 17 |
| Chemical | Nov 19 |
| Plasma | Dec 09 |

Table 4-8: Dates when the drop tests were made for Aluminum 2017

| Treatment | Fowkes [mJ/m ²] | |
|-------------------|-----------------------------|----------------------|
| | Total energy | Dispersive component |
| Without treatment | 51,18282 | 41,6632 |
| Abrasing (80) | 61,7919 | 40,7153 |
| Abrasing (100) | 60,536 | 42,1666 |
| Chemical | 71,8681 | 45,5722 |
| Plasma | 74,7044 | 47,6689 |

Table 4-9: Sum of the Fowkes results of Aluminum 2017

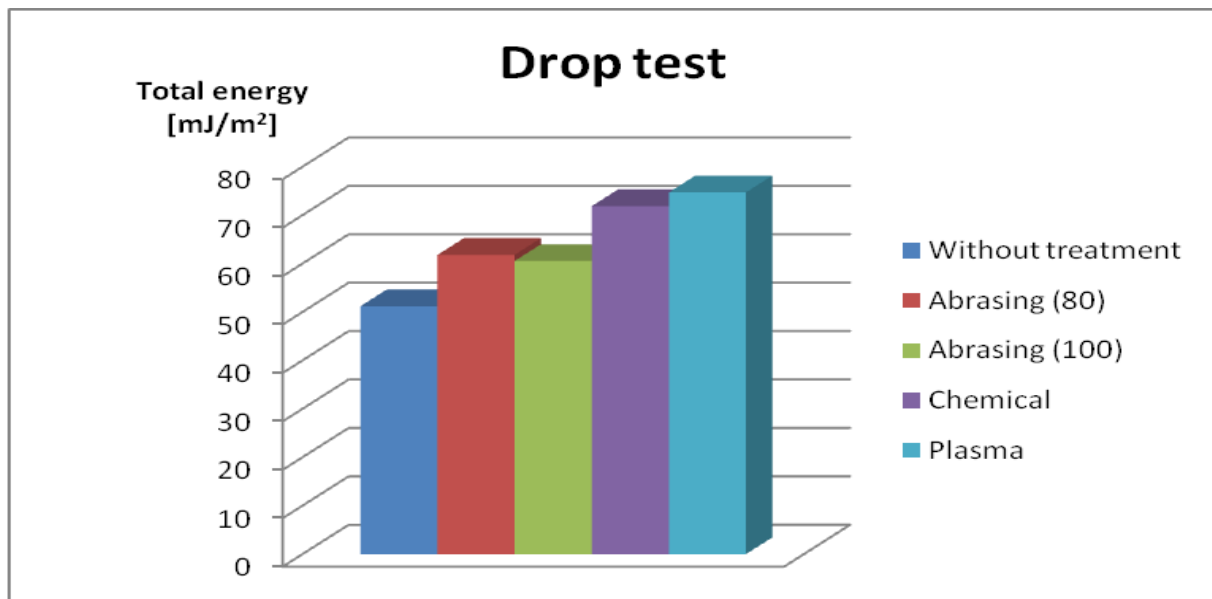


Figure 4-4: Diagram of the total energy by Fowkes method of Aluminum 2017

For the aluminum, the results from the dispersive component do not differ so much from the initial. The two different abrasion methods (100 and 80) have almost the same results. The total energy is a little bit higher for abrasion 80 but the dispersive component is a little bit higher for the abrasion 100. So we decided to try the abrasion 100 again for the next test, the shear strength test. This treatment with the chemical was the two treatments that were decided to be tested for the shear strength tests. But like you also can see the plasma treatment was included in the different tests. This

was not the plan from the beginning but after some more researches and advice from the plasma company and the factor that the plasma machine was available, it became one of the four treatments for the drop tests and also one of the best ones and will therefore also be chosen to the shear strength tests.

Stainless steel 316L

See appendix 7 for exact values from all the tests.

| Drop test | Date (2010) |
|-------------------|-------------|
| without treatment | Nov 15 |
| Abrasion (80) | Nov 17 |
| Abrasion (100) | Nov 17 |
| Chemical | Nov 24 |
| Plasma | Dec 09 |

Table 4-10: Dates when the drop tests were made for Stainless steel 316L

| Treatment | Fowkes [mJ/m ²] | |
|-------------------|-----------------------------|----------------------|
| | Total energy | Dispersive component |
| Without treatment | 55,9367 | 39,8942 |
| Abrasing (80) | 57,8962 | 39,5262 |
| Abrasing (100) | 63,627 | 41,6945 |
| Chemical | 77,1341 | 50,0184 |
| Plasma | 75,1003 | 49,0838 |

Table 4-11: Sum of the Fowkes results of Stainless steel 316L

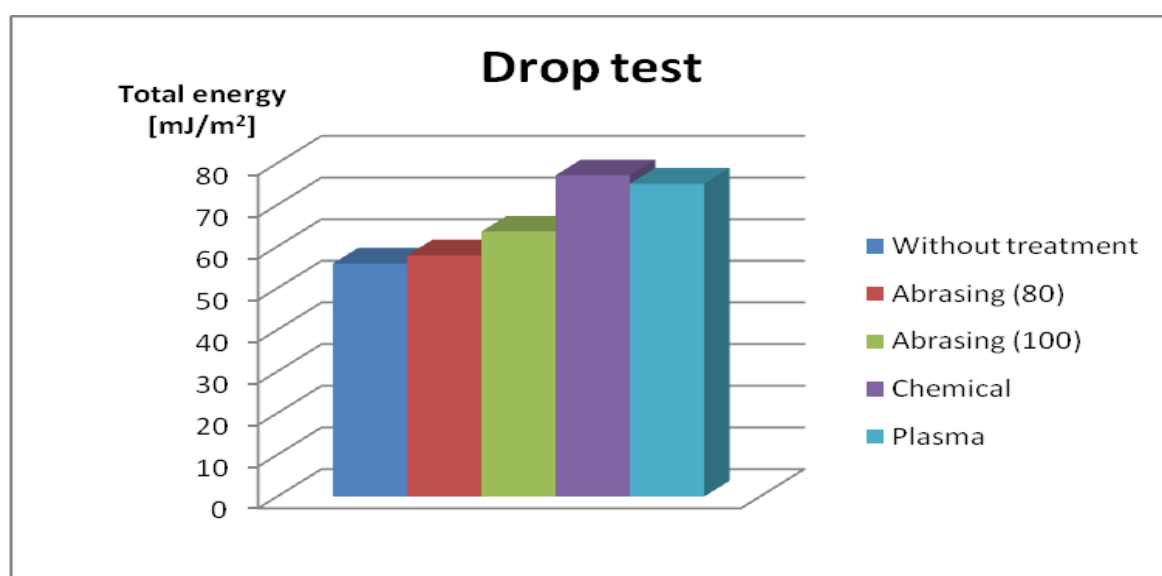


Figure 4-5: Diagram over the total energy by the Fowkes method of Stainless steel 316L

The chemical treatment is almost 10 units better than the abrasion (100) so it should maybe be chosen as the preferred treatment. But abrasion is a much easier and cheaper method to work with and also much more environment-friendly than the chemical and therefore the both treatments will be tested for the shear strength test. The plasma treatment, that was explained earlier, came in late as a treatment for the metals but has still a good result and will also be tested for the shear strength tests.

4.1.3 Shear strength test results

After the drop test result we were able to choose the three best surface treatments for Aluminum and Stainless Steel and the two best surface treatments for Carbon/PEEK, Carbon/PPS and Glass/Pa12.

For Aluminum and Stainless Steel we chose abrasion, chemical and plasma treatment. For Carbon/PEEK, Carbon/PPS and Glass/Pa12 it is flame and plasma treatment.

In the first part of the shear strength test we bond the materials among themselves. That means for example Aluminum with Aluminum, Carbon/PEEK with Carbon/PEEK and so on.

To find out which is the best surface treatment we chose one structural adhesive, an epoxy adhesive, which has good mechanical properties and also high temperature resistance. The chosen adhesive for this part of the test is the Scotch-Weld DP 490 from the company 3M.

The adhesive bonding process is explained later in the “finding the best adhesive” part. In this part there will also be the technical information about the adhesive.

In table 4.12 you can see the results of the shear strength test. The first column comprised the tested material and the combinations. In the following columns you can see the treatment, the test number, the maximal stress, the standard deviation, the average of the maximal stress and in the end the failure mode. The standard deviation gives a clue if the measurement was good or not. The smaller the number is the better was the test. In section 3.6, “possible bond failures”, you can find the explanations about the different possible failure modes. Due to late deliveries of composites samples we did not have enough samples to do the right amounts of tests. Therefore there are sometimes only two shear strength tests made. The best solution would be to do more than three tests for each combination. Like this the standard deviation would be lower because the results would not vary so much.

| Material | Treatment | No. | Max stress | Std deviation | Average | Failure mode |
|---------------------------------------------------|-----------|-----|------------|---------------|-----------|--------------|
| Aluminium <i>with</i> Aluminium | Abrasion | 1 | 16,67 MPa | 2,13 | 18,82 MPa | cf |
| | | 2 | 18,87 MPa | | | cf |
| | | 3 | 20,93 MPa | | | cf |
| | Chemical | 1 | 22,92 MPa | 2,02 | 20,60 MPa | cf |
| | | 2 | 19,66 MPa | | | cf |
| | | 3 | 19,22 MPa | | | cf |
| | Plasma | 1 | 17,28 MPa | 2,11 | 18,77 MPa | if |
| | | 2 | 20,26 MPa | | | acf |
| Stainless Steel <i>with</i> Stainless Steel | Abrasion | 1 | 24,49 MPa | 3,94 | 20,84 MPa | cf |
| | | 2 | 21,38 MPa | | | cf |
| | | 3 | 16,66 MPa | | | cf |
| | Chemical | 1 | 21,23 MPa | 0,59 | 21,91 MPa | cf |
| | | 2 | 22,17 MPa | | | cf |
| | | 3 | 22,32 MPa | | | cf |
| | Plasma | 1 | 25,05 MPa | 1,48 | 24,05 MPa | acf |
| | | 2 | 23,05 MPa | | | 70%cf+30%af |
| | | | | | | |
| Glass/Pa12 <i>with</i> Glass/Pa12 | Flame | 1 | 18,93 MPa | 3,10 | 21,12 MPa | af90% |
| | | 2 | 23,31 MPa | | | af80% |
| | Plasma | 1 | 14,53 MPa | 0,65 | 14,07 MPa | af50% |
| | | 2 | 13,61 MPa | | | af50% |
| Carbon/PEEK <i>with</i> Carbon/PEEK | Flame | 1 | 14,95 MPa | 5,65 | 17,29 MPa | af60% |
| | | 2 | 23,73 MPa | | | cf |
| | | 3 | 13,18 MPa | | | af50% |
| | Plasma | 1 | 17,04 MPa | 3,00 | 14,92 MPa | af |
| | | 2 | 12,80 MPa | | | af |
| | | | | | | |
| Carbon/PPS <i>with</i> Carbon/PPS | Flame | 1 | 18,55 MPa | 3,02 | 15,07 MPa | df50% |
| | | 2 | 13,32 MPa | | | df100% |
| | | 3 | 13,33 MPa | | | af90% |
| | Plasma | 1 | 18,37 MPa | 1,92 | 16,90 MPa | df50% |
| | | 2 | 17,59 MPa | | | af80% |
| | | 3 | 14,73 MPa | | | af50% |

Table 4-12: Results from shear strength test to find the best surface treatment

Aluminum – Aluminum

The difference between abrasion and chemical treatment is only 1,78 MPa. That is why we chose abrasion as a treatment although the chemical treatment got a higher result. The abrasion is finally cheaper than the chemical treatment because the process is faster and the needed tools are also cheaper. You need therefore only sandpaper, but for the chemical treatment you need different kinds of chemical products and you have to keep special security policies. It is the same case for the

plasma treatment. The result is almost the same like abrasion, but the plasma machine is more expensive and there are also stricter security policies for the plasma than for the abrasion.

Stainless steel – Stainless steel

For the stainless steel we chose also the surface roughening (abrasion) because of the cheaper process. The failure is like the aluminum joint, a cohesive failure. The plasma however has an adhesive failure, which is not so good. For that reason and because we only had the machine the week when we did not have enough samples and that the ordered adhesives were delayed, we decided to do the abrasion.

Glass/Pa12 – Glass/Pa12

The best stress result was 21.12 MPa with the flame treatment; therefore we chose the flame treatment as the best choice. The plasma treated joint only achieved 14.07 MPa. In both types of treatments there is an adhesive failure.

Carbon/PEEK – Carbon/PEEK

Looking at the Carbon/PEEK you can see a big difference between the results for the flame treatment but also for the plasma treatment. The explanation for this could be the difficulty to reproduce the same surface treatment to the different samples. Finally we chose also the Flame treatment, because it got the better result and it is also cheaper and easier to accomplish.

Carbon/PPS – Carbon/PPS

For the Carbon/PPS the plasma treated joint achieved 16.9 MPa and the flame treated joint 15.07 MPa. The differences between them is though 1.83MPa and not so huge. Regarding the costs and the expense of the plasma treatment it is finally more effective to use the flame treatment.

4.2 Finding the best adhesive

In this, next step we want to find the best adhesive. In section 4.1.3 we already chose the best surface treatment which is going to be used for the following process.

To find the best adhesive we decided to choose the three best adhesives we can find and try them by doing the shear strength test. To be able to evaluate the result we will bond the material 3 times among themselves, that means Aluminum with Aluminum, Carbon/PEEK with Carbon/PEEK and so on.

The only adhesive groups which are interesting for the project are Acrylic, Epoxy and Polyester because they have good tensile shear strength properties, are heat resistant and are used for metals and also plastics at the same time. The others are either not heat resistant or suitable for metals and plastics.

So we contacted some different suppliers which offer this kind of structural adhesives. The contacted companies are the follows:

| Company | Adress | Name | Telephone number |
|---------------------|------------------------------------------------------------------|---------------------------|----------------------|
| Henkel-Loctite | www.henkel.com | | 0033 16 41 77 000 |
| Loctite | www.loctite.de | | 0049 89 320 800 1600 |
| Eleco Produits | www.eleco-produits.fr | Pascal Arnaudin | |
| Hunstmann | www.huntsmann.com | Nadine Petitot, Katy Rost | 0044 12 23 83 21 21 |
| 3M | www.mmm.com | Thierry | 0033 13 03 16 264 |
| ITW Plexus | www.itwplexus.com | | |
| ATP Adhesive System | www.atp-ag.com | | |
| Polytech PT Germany | www.polytec-pt.de | Schobert | 0049 7243 604 400 |
| Polytech France | | Stephan Tardy | 0033 14 96 56 900 |
| Kremer | | | 0049 75 65 91 120 |
| Final | www.final.fr | | 0033 38 85 42 600 |

Table 4-13: Adhesive company that where contacted

The only company which answered my first mail was 3M. So finally tried to call all companies personally to get more information about the adhesive they offer. So now it was important to search the best adhesive of all the suggested possibilities. This happened by comparing the characteristics e.g. the operating temperature and so on.

The final chosen adhesives are listed below:

| Name | DP490 | Duralco 4525 | EPO-TEK 353 ND-T | Hysol 9466 | H3101 |
|-------------------------|--------------------------------------------|--------------------------------------|---------------------------------------|----------------------------------|----------------------------------|
| Company | 3M Scotch-Weld | Polytec PT | EPOXY-Technologys | Loctite | Loctite |
| Type | Epoxy | Epoxy | Epoxy | Epoxy | Methacrylate |
| Number of components | 2 | 2 | 2 | 2 | 2 |
| Mixing ratio | 100:50 | 100:8 | 10:1 | 100:50 | 1:1 |
| Color | | | | | |
| Part A | black | black | Tan | white paste | Crema |
| Part B | white | amber | Amber | liquid white | pale yellow |
| Viscosity [cPs] | | 25 500 | 9.000-15.000 | | 45.000-110.000 |
| Cure time | 2-24h at 23°C +60min at 80°C | 16h at 23°C 10min at 80°C | 60min at 150°C | | 20-25 min |
| pot life | 3h | 30min | 3h | 60min | |
| shelf life | | 6 month @ RT | 12 month @ RT | | +2°C to + 4°C |
| Shore D Hardness | - | D90 | D80 | D60 | |
| Operating Temp. | | | | | |
| Continuous | 120°C | | -55°C to 225°C | | |
| Intermittent | | 260°C | -55°C to 325°C | | |
| used for this materials | PEEK, PPS, Pa12, Aluminum, Stainless Steel | PEEK, PPS, Aluminum, Stainless Steel | PEEK, PPS, Aluminium, Stainless Steel | Pa12, Aluminium, Stainless Steel | Pa12, Aluminium, Stainless Steel |

Table 4-14: The chosen adhesive

Bonding procedure

After the first steps: 'clean', 'dry' and 'surface treatment', the treated area has to be bordered by tape. The whole shape of the sample is 25 mm x 150 mm. The surface where the adhesive can be applied has to be 312,5 mm. The surface should therefore have a shape like 12,5 mm x 25 mm. In our tests the adhesives was applied directly after the surface treatment.

The adhesive was mixed by weight in a small aluminum bowl. The mixing ratio is listed in the table above. The amount of adhesive we used was about 4-10 gram. 1 gram of 'DP490' for example is enough to cover 6 surfaces or 5 gram of the 'EPO TEK 353ND-T' is enough to cover 24 surfaces.

To be able to calibrate the joints with the same thickness we added glass balls with a thickness of 0.2mm. The amount of the glass balls is 1% of the total weight of the adhesive. When the two parts of the adhesive and glass balls are in the bowl they have to be good agitated with a scraper or something similar.

After the mixing, the adhesive was applied directly with the scraper to the treated surface. When the adhesive is spread over both surfaces the two samples were put together. With two brackets it was secured that the samples do not move any more until they are cured.

For the 'DP490' and the 'Duralco 4525' we waited at least 16 hours until the next day. Then we put the samples in the oven to polymerise the adhesive. The 'DP490' has to be in the oven for 60min at 80°C and the 'Duralco 4525' 10min at 80°C. The 'EPO TEK 353ND-T' do not need to cure at the air and

can be put directly in the oven. We decided to put it 60min at 150°C to reach the same properties like on the data sheet.

After the curing and the polymerisation in the oven we waited approximated 15 minutes until the samples were cooled down to room temperature. Then we started to do the shear strength test with two different machines, depending on which machine was available and function-able. The two machines were, '3R RP50 ELP22PG' and 'Istron 4204'. The results of the shear strength test are shown in the table below.

| Material | Adhesive | No | Max stress | Std. deviation | Average | Failure mode |
|---------------------------------------------------------------------------------------|--------------------|----|------------|----------------|-----------|---------------|
| Aluminium (Abrasion) <i>with</i> Aluminium (Abrasion) | DP490 | 1 | 16,67 MPa | 2,13 | 18,82 MPa | cf |
| | | 2 | 18,87 MPa | | | cf |
| | | 3 | 20,93 MPa | | | cf |
| | Duralco 4525 IP | 1 | 12,27 MPa | 0,38 | 12,00 MPa | af30%+af70% |
| | | 2 | | | | error |
| | | 3 | 11,73 MPa | | | af100% |
| | EPO TEK 353ND-T | 1 | 15,17 MPa | 0,49 | 14,94 MPa | afa80%+afb20% |
| | | 2 | 15,28 MPa | | | afa50%+afa50% |
| | | 3 | 14,38 MPa | | | afa90%+afb10% |
| Stainless Steel (Abrasion) <i>with</i> Stainless Steel (Abrasion) | DP490 | 1 | 24,49 MPa | 3,94 | 20,84 MPa | cf |
| | | 2 | 21,38 MPa | | | cf |
| | | 3 | 16,66 MPa | | | cf |
| | Duralco 4525 IP | 1 | 19,57 MPa | 0,45 | 19,41 MPa | af90%+af10% |
| | | 2 | 19,76 MPa | | | af100% |
| | | 3 | 18,91 MPa | | | af100% |
| | EPO TEK 353ND-T | 1 | 19,24 MPa | 0,60 | 19,61 MPa | afa20%+afb80% |
| | | 2 | 20,31 MPa | | | afa20%+afb80% |
| | | 3 | 19,29 MPa | | | af80%+afb20% |
| Carbon/PEEK (Flame) <i>with</i> Carbon/PEEK (Flame) | DP490 | 1 | 14,95 MPa | 5,65 | 17,29 MPa | 60%af+40%af |
| | | 2 | 23,73 MPa | | | cf |
| | | 3 | 13,18 MPa | | | afa50%+afb50% |
| | Duralco 4525 IP | 1 | 14,84 MPa | 1,49 | 13,99 MPa | df50% |
| | | 2 | 11,94 MPa | | | af10%+af90% |
| | | 3 | 12,78 MPa | | | df50%+df50% |
| | EPO TEK 353ND-T | 1 | 13,88 MPa | 0,88 | 14,79 MPa | df70%+df30% |
| | | 2 | 14,86 MPa | | | df50%+df50% |
| | | 3 | 15,63 MPa | | | af10%+af90% |
| Carbon/PPS (Flame) <i>with</i> Carbon/PPS (Flame) | DP490 | 1 | 18,55 MPa | 3,02 | 15,07 MPa | df50%+df50% |
| | | 2 | 13,32 MPa | | | df100%+df0% |
| | | 3 | 13,33 MPa | | | afa90%+afb10% |
| | Duralco 4525 IP | 1 | 16,53 MPa | 1,31 | 17,49 MPa | df50%+df50% |
| | | 2 | 16,95 MPa | | | df50%+df50% |
| | | 3 | 18,98 MPa | | | df50%+df50% |
| | EPO TEK 353ND-T | 1 | 20,41 MPa | 3,64 | 17,43 MPa | afa30%+afb70% |
| | | 2 | 13,38 MPa | | | afa90%+afb10% |
| | | 3 | 18,50 MPa | | | afa60%+afb40% |

| Material | Adhesive | No | Max stress | Std. deviation | Average | Failure mode |
|-----------------------------------------------------------------------|------------|----|------------|----------------|-----------|----------------------------|
| Glass/Pa12 (Flame) <i>with</i> Glass/Pa12 (Flame) | DP490 | 1 | 18,93 MPa | 3,10 | 21,12 MPa | af90%+af10% af80%+af20% |
| | | 2 | 23,31 MPa | | | |
| | H3101 | 1 | | | | |
| | | 2 | | | | |
| | | 3 | | | | |
| | Hysol 9466 | 1 | | | | |
| | | 2 | | | | |
| | | 3 | | | | |

Table 4-15: Result from shear strength test to find the best adhesive

Unfortunately we were not able to finish the Glass/Pa12 test because of a lack of samples. They were ordered in the beginning of the project, but did not arrive until the end of the project.

To decide now which adhesive that is the best one to bond the composites with the metals, we have to consider the combinations.

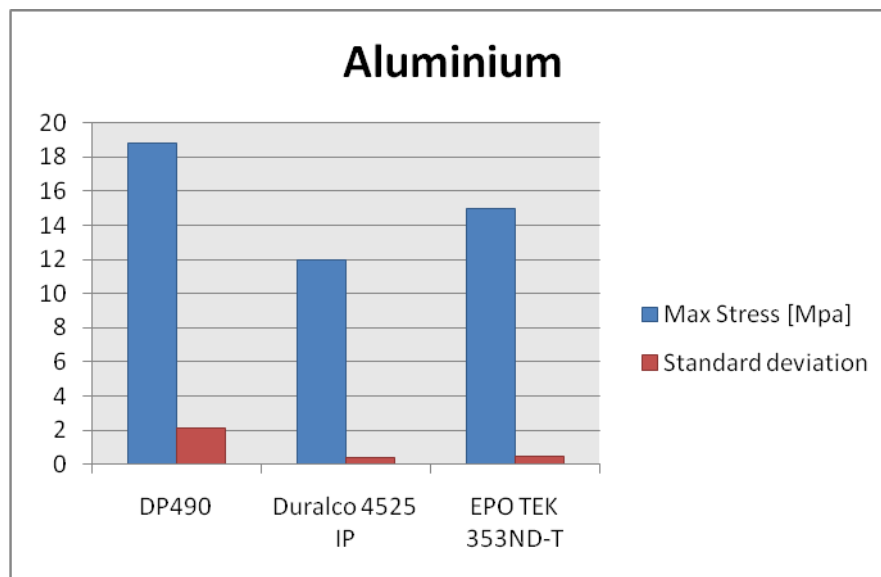


Figure 4-6: Max shear strength and standard deviation of aluminum

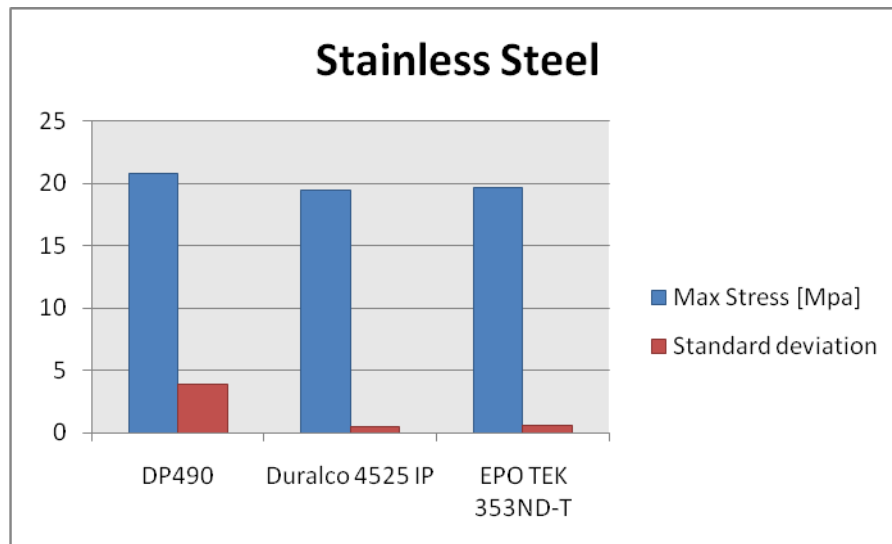


Figure 4-7: Max shear strength and standard deviation of stainless steel

For Carbon/PEEK with Aluminum and Carbon/PEEK with Stainless Steel we decided to use the DP490. Both Aluminium and Stainless Steel have a cohesive failure with the DP490 which is the best failure because the most stress is in the adhesive. In addition the DP490 achieved the highest strength. For the Aluminium the standard deviation is not so good, but even the lowest result of the three test numbers is still higher than the results for the other adhesives. For the Stainless Steel all the results are about 20MPa so you could also choose another one. But in combination with the Carbon/PEEK we chose the DP490. The best results for the Carbon/PEEK were with the DP490. Regarding the average we chose the DP490. But this result have a high standard deviation and therefore it would be good to do more tests to insure that it is the right one, but we did not have enough samples to do that.

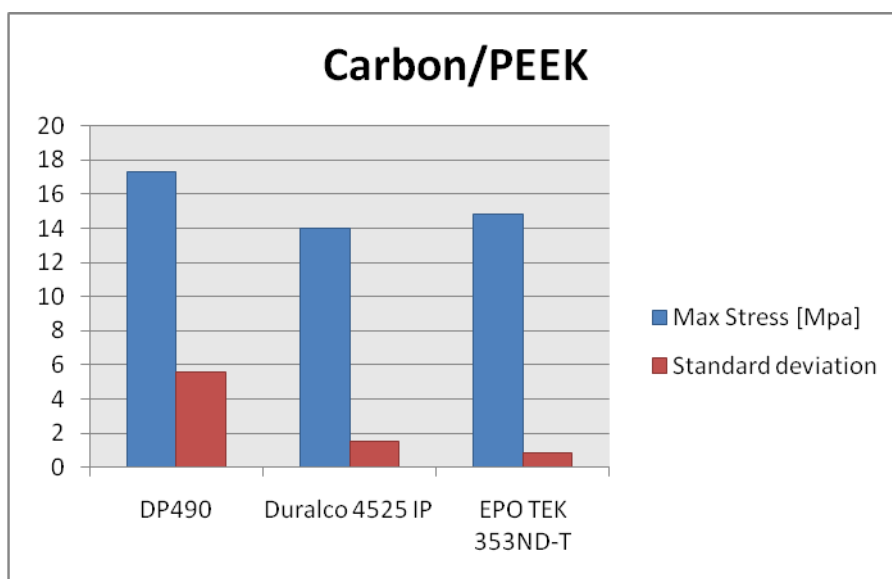


Figure 4-8: Max shear strength and standard deviation of Carbon/PEEK

For the combination Carbon/PPS with Aluminum we chose the DP490 because the two other adhesives for Aluminum are not so good. And in addition the differences between the results for the PPS are not so high.

Regarding the results for the PPS, the 'Duralco' and the 'EPO TEK' achieved 17MPa whereas the 'DP490' achieved only 15MPa. Regarding the standard deviation the results for the 'Duralco' are better because the standard deviation is lower. The results for the Stainless steel however are more or less the same with approximately 20MPa. That is why we chose the 'Duralco 4525 IP' for the combination Carbon/PPS with Stainless Steel.

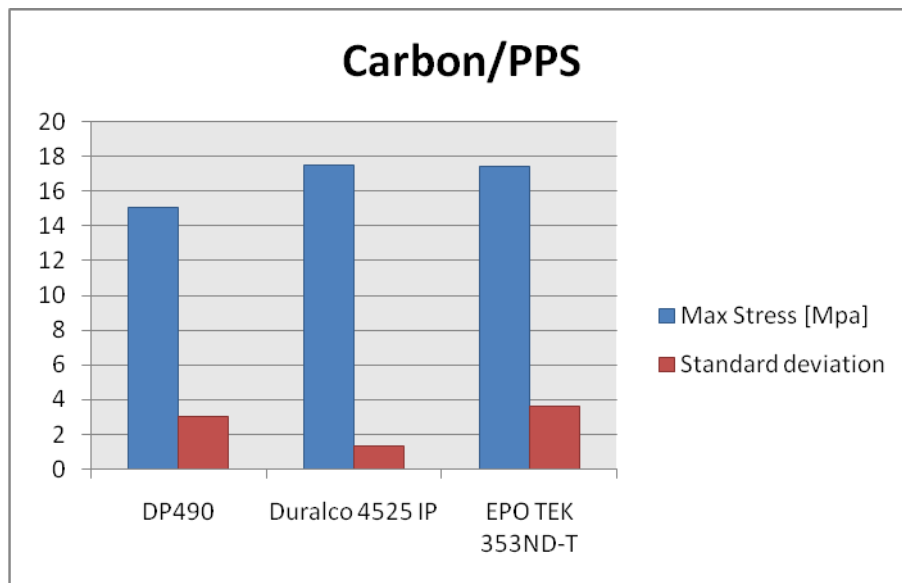


Figure 4-9: Max shear strength and standard deviation of Carbon/PPS

4.3 Test of best choice

| Material | Adhesive | Treatment | No. | Max stress | Std. deviation | Average | Failure mode |
|----------------------------------------|-----------------|-----------|-----|------------|----------------|-----------|---------------|
| Carbon/PEEK Aluminium | DP490 | nothing | 1 | 6,19 MPa | | 6,19 MPa | afa0%+afb100% |
| Carbon/PEEK with Aluminium | DP490 | Flame | 1 | 12,60 MPa | 2,785 | 15,40 MPa | afa100%+afb0% |
| | | | 2 | 18,17 MPa | | | afa100%+afb0% |
| | | Abrasion | 3 | 15,44 MPa | | | afa100%+afb0% |
| Carbon/PEEK Stainless Steel | DP490 | nothing | 1 | 6,95 MPa | | 6,95 MPa | afa0%+afb100% |
| Carbon/PEEK with Stainless Steel | DP490 | Flame | 1 | 18,19 MPa | 3,770 | 22,44 MPa | afa100%+afb0% |
| | | | 2 | 25,38 MPa | | | 20%cf+80%af |
| | | Abrasion | 3 | 23,75 MPa | | | 20%cf+80%af |
| Carbon/PPS Aluminium | DP490 | nothing | 1 | 15,85 MPa | | 15,85 MPa | afa0%+afb100% |
| Carbon/PPS with Aluminium | DP490 | Flame | 1 | 23,73 MPa | 1,180 | 23,24 MPa | cf |
| | | | 2 | 21,89 MPa | | | 70%cf+30%df |
| | | Abrasion | 3 | 24,09 MPa | | | cf |
| Carbon/PPS Stainless Steel | Duralco 4525 IP | nothing | 1 | 11,52 MPa | | 11,52 MPa | afa0%+afb100% |
| Carbon/PPS with Stainless Steel | Duralco 4525 IP | Flame | 1 | 16,61 MPa | 1,762 | 16,01 MPa | afa0%+afb100% |
| | | | 2 | 17,40 MPa | | | afa0%+afb100% |
| | | Abrasion | 3 | 14,03 MPa | | | afa0%+afb100% |

Table 4-16: Table of chosen treatment and adhesive with initial value

This table shows the results of the final bonding between the composites and the metals, and the shear strength of the combinations without any surface treatment to be able to compare the results. The tests without treatment were only made once for each combination because there were not enough samples. The results show that it is a big difference between a treated surface and an untreated surface. It also shows that it is easier to bond PPS than PEEK with the metals due to PPS's high initial value without any surface treatment.

For Carbon/PEEK with aluminum the average shear strength is 15,40 MPa with an adhesive failure on the Carbon/PEEK. Comparing with the results from table 4.15 (finding the best adhesive), Carbon/PEEK with Carbon/PEEK had an average strength of 17,29 MPa but with a high standard deviation. Aluminum with aluminum had 18,82 MPa in strength with a cohesive failure.

The Carbon/PEEK with stainless steel have the average strength of 22,44 MPa but also with the highest standard deviation mostly due to the low result of the first test that only had 18,19 MPa in

strength. The reason may be that it had an 100% adhesive failure while the others with higher results had also a part with a cohesive failure.

The strength of Carbon/PEEK with Carbon/PEEK is like before 17,29 MPa with a very high standard deviation were the lowest value is 13,18 MPa, with an adhesive failure, and the highest value is 23,73 MPa, with a cohesive failure. The big difference may be due to the surface treatment, in this case the flame treatment. The time and speed the samples passing the flame are very important. The stainless steel with stainless steel strength is 20,84 MPa which is quite close to the result of Carbon/PEEK with stainless steel.

The Carbon/PPS with aluminum have a very high initial shear strength of 15,85 MPa, without treatment. This means that it is easy to bond these materials. It has 23,24 MPa with the surface treatment, which is the best result of all the combinations and also with the best standard deviation and a cohesive failure.

The results of aluminum with aluminum have 18,82 MPa and Caron/PPS with Carbon/PPS have 15,07 MPa. The both differ quite much from Carbon/PPS with aluminium and that can have many reasons, e.g. the surface treatment that is not made in the same ways. For example, the Carbon/PPS is more “burned” and the aluminium has deeper roughness after the abrasion.

The Carbon/PPS with stainless steel have a shear strength of 16,01 MPa with a adhesive failure on the stainless steel. The results from table 4.15 on the Carbon/PPS with Carbon/PPS is 17,49 MPa with a df failure and stainless steel with stainless steel is 19,41 MPa. So it is normal that all the adhesive stays on the composite with the combination of Carbon/PPS with stainless steel.

5 FINAL MATRIX

| Material | Treatment | Adhesive | Initial | Max Stress | St deviation | Failure Mode |
|--------------------------------|-------------------|-----------------|-----------|------------|--------------|---------------|
| Carbon/PEEK Aluminium | Flame Abrasion | DP490 | 6,19 MPa | 15,40 MPa | 2,785 | afa100%+afb0% |
| Carbon/PEEK Stainless Steel | Flame Abrasion | DP490 | 6,95 MPa | 22,40 MPa | 3,77 | 20%cf+80%af |
| Carbon/PPS Aluminium | Flame Abrasion | DP490 | 15,85 MPa | 23,24 MPa | 1,18 | cf |
| Carbon/PPS Stainless Steel | Flame Abrasion | Duralco 4525 | 11,52 MPa | 16,01 MPa | 1,762 | afa0%+afb100% |

Table 5-1: Final matrix of chosen treatment and adhesive

The final matrix shows the summary of the best results from all tests that were made.

6 CONCLUSION

For most of us this has been the first time to be in contact with a real project and to use Project Management tools. We gratefully discovered that there are a lot of powerful tools available to manage a project successfully, like Gantt chart, Pert chart, WBS etc.

We find out that a good and successful project does not start asking for the raw materials or switching on the pilot plant machines. A success project start far earlier, in a meeting room, organizing and pinpointing every task that should be done during the whole project, selecting the teamwork, the feasibilities, the deadlines, the monitoring process, the risks, the budget, the scope...

From the very beginning of this project we have been creating and using this project management tools, trying to define all the tasks, the milestones and establishing the deadlines for each of these tasks.

During the first part of the project we have been following the planning but as it's normal the things never go one hundred percent well. We had problems with the adhesive suppliers and their delivers. We got the materials approximately 3 weeks later than we expected. That means a big delay considering that the whole project is only 14 weeks. Finally we had to change the planning and leave the final stability studies and tests, which we wanted and planned to do, but besides that we consider that it have been a very successful project planning.

The aim of this project regarding to the technical part was to find out the best bonding process to assemble 3 different thermoplastic matrix composites with 2 different metals.

| | | |
|-------------|-----|-----------------|
| Carbon/PEEK | <-> | Stainless steel |
| Carbon/PEEK | <-> | Aluminum |
| Carbon/PPS | <-> | Stainless steel |
| Carbon/PPS | <-> | Aluminum |
| Glass/Pa12 | <-> | Stainless steel |
| Glass/Pa12 | <-> | Aluminum |

Finally we were able to solve this problem. The best surface treatments for all the materials were worked out and the three adhesives for the different combinations were also founded out. For the combinations PEEK/PPS with aluminum/stainless steel the best adhesive were chosen by doing the shear strength test and the required shear strength stress were achieved.

For the combination Pa12 with aluminum and stainless steel it was not possible to find out the best adhesive because the test could not be continued. Like before suspected there were problems with the delivery of samples and therefore the tests of Pa12 could not be finished in time.

Another problem was the contact with adhesive suppliers. Most suppliers did not answer to mails at all so all of them were contacted again by telephone. Once again it took some time to choose the three best adhesives. After the choice of the three adhesives to test, it took further weeks to get the adhesives from the suppliers. This adhesive ordering process took finally so much time, that the stability test could not be done before the Christmas holidays and finally not at all.

In the next project it is important to solve problems, where you are depended on suppliers, with priority. So the first step would have been the ordering of the adhesives directly by telephone without the laborious way via mail.

To finish the project the three different adhesives, which are already arrived, should be applied to the Pa12-Pa12 combination to find out the best adhesive. When the best adhesive of the three is chosen it can be bonded with the aluminum and stainless steel to find out the final shear strength stress.

After this, the bonded material, of all the combinations, with the best treatment and the best adhesive has to be checked in salt spray, temperature and humidity chambers to ensure that the bonding is environment resistant.

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10 APPENDIX

10.1 Appendix 1 – Requirements document

Requirement Document

TECHNACOL REQUIREMENTS

Topic:

1. Definition of bonding technologies to assemble thermoplastic matrix composites (TPMC) with metals. Materials: Carbon PEEK, Carbon PPS, Glass Pa12 with Aluminum and Stainless Steel
 - a. Surface preparation research + application
 - b. Adhesive/glue research + application
 - c. Flexibility on applications time. A process that allows the factory to have some extra time
 - d. between the different process steps
 - e. Stability: In terms of temperature and corrosion
 - f. Strong enough (at least 15-20 MPa)
 - g. Should be a manual process
 - h. A semi-automatic or automatic scale up should be feasible
 - i. Environment care
2. Other bonding procedures
 - a. Easier
 - b. Faster: Decrease the automatic process
 - c. Special treatments to increase the speed of the bonding process
3. Study the possibilities of semi-automatic processes:
 - a. Basic mock-up sketch
 - b. Applications
 - c. Equipments
 - d. Cost

Deliverables

1. Surface preparation protocol
2. Adhesive/glue application protocol
3. Stability studies protocols
4. Stability results reports
5. Other bonding procedures protocols
6. General manufacturing procedure protocol
7. A semi-automatic scale up procedure protocol: Equipments, devices, cost evaluation

ENIT REQUIREMENTS

1. Intermediate project review and report
2. Final presentation and report
3. The project planning
4. The Scope of the project

OUR REQUIREMENTS

1. Total access to the lab facilities and documentation. In a reasonable schedule
2. We need a continues good theoretical and technical assistance

10.2 Appendix 2 – Sample amount

For the drop test

| DROP TEST | | | |
|-----------|---------|---------------------|----------------------------|
| Material | initial | Surface preparation | Total samples for the test |
| PPS | 1 | 10 | 11 |
| PEEK | 1 | 10 | 11 |
| Pa12 | 1 | 10 | 11 |
| Aluminium | 1 | 2 | 3 |
| SS | 1 | 2 | 3 |

We will test the bonded materials at least three times to get an average.

1-Sandblasting (3 dif.sands x2 just in case)
2-Paper abration
3- Corona
4- Flame
5-Plasma

1-Sandblasting
2-Paper abration
3- Corona
4- Flame
5-Plasma

1- chemical threatment
2- Paper abration

1- chemical threatment
2- Paper abration

1-Sandblasting
2-Paper abration
3- Corona
4- Flame
5-Plasma

For the bonding process

| BONDING PROCESS STUDY | | | | | | | |
|-----------------------|-------------------------------|----------|-------|---------------------------|-----------------|---------------|--------------------------------------------------|
| Composite | Composite Surface preparation | Adhesive | metal | Metal Surface preparation | Reproducibility | Total samples | Conclusion |
| PPS | 5 | 1 | 1 | 2 | 3 | 30 | Best Surface preparation for PPS and for metals |
| PEEK | 5 | 1 | 1 | 2 | 3 | 30 | Best Surface preparation for PEEK and for metals |
| Pa12 | 5 | 1 | 1 | 2 | 3 | 30 | Best Surface preparation for Pa12 and for metals |

1-Sandblasting
2-Paper abration
3- Corona
4- Flame
5-Plasma

In this step the adhesive is not relevant. We will test just one epoxi.

We are going to test the composites with only one metal (with two different surface preparations) taking into account that the behaviour of the two methals will be the same.

We will test the bonded materials at least three times to get an average.

1-Sandblasting
2-Paper abration
3- Corona
4- Flame
5-Plasma

1-Sandblasting
2-Paper abration
3- Corona
4- Flame
5-Plasma

1- chemical threatment
2- Paper abration

After this process we will get the best surfaces preparation for each component.

Now, according to the results, we will look for the best adhesive:

The composite surface preparation has been selected on the above process

We are going to test the adhesives with the two different metals

The metal surface has been selected on the above process

| Composite | Composite Surface preparation | Adhesive | metal | Metal Surface preparation | Reproducibility | Total samples | Conclusion |
|-----------|-------------------------------|----------|-------|---------------------------|-----------------|---------------|------------------------|
| PPS | 1 | 3 | 2 | 1 | 3 | 18 | Best adhesive for PPS |
| PEEK | 1 | 3 | 2 | 1 | 3 | 18 | Best adhesive for PEEK |
| Pa12 | 1 | 3 | 2 | 1 | 3 | 18 | Best adhesive for Pa12 |

In this step the adhesive is the most important thing. We will make the bonding process using three different adhesives. Hopefully they will be two epoxis and one silicone. But it will depends on the supplier advices.

We will test the bonded materials at least three times to get an average.

For this last process we will need: 27 samples of Aluminium and Stainless steel

For the stability process

1 sample added to aluminium and 1 added to SS (x3 reproducibility)

3 samples added to PEEK, 3 samples added to PPS and 3 samples added to Pa12

We will test three times and take the average of the mesures

3 samples added to aluminium and 3 added to SS

We will test three times and take the average of the mesures

3 samples added to PEEK, 3 samples added to PPS and 3 samples added to Pa12

| STABILITY PROCESS | | | | | |
|-------------------|------|-----|------|-----------|-----------------|
| | PEEK | PPS | Pa12 | Aluminium | Stainless steal |
| Salt spray | | | | | |
| initial | 6 | 6 | 6 | 9 | 9 |
| final | 6 | 6 | 6 | 9 | 9 |
| Temperature | | | | | |
| initial | 0 | 0 | 0 | 0 | 0 |
| final | 6 | 6 | 6 | 9 | 9 |
| Humidity | | | | | |
| initial | 0 | 0 | 0 | 0 | 0 |
| final | 6 | 6 | 6 | 9 | 9 |
| total samples | 24 | 24 | 24 | 36 | 36 |

10.3 Appendix 3 – Carbon/PEEK drop test results

10.3.1 Carbon/PEEK – Without treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | PEEK | 69.4 | 65.8 | 67.6 | 0.381 | manul |
| 2 | Glycérol | PEEK | 71.5 | 68.9 | 70.2 | 0.339 | manul |
| 3 | Glycérol | PEEK | 66.4 | 64.8 | 65.6 | 0.413 | manul |
| 4 | Formamide | PEEK | 57.5 | 53.6 | 55.6 | 0.566 | manul |
| 5 | Formamide | PEEK | 55.9 | 54.7 | 55.3 | 0.569 | manul |
| 6 | Formamide | PEEK | 53.1 | 52.3 | 52.7 | 0.606 | manul |
| 7 | Diiodométhane | PEEK | 24.0 | 23.2 | 23.6 | 0.916 | manul |
| 8 | Diiodométhane | PEEK | 28.0 | 26.4 | 27.2 | 0.889 | manul |
| 9 | Diiodométhane | PEEK | 28.1 | 27.4 | 27.8 | 0.885 | manul |
| 10 | DMSO | PEEK | 23.5 | 21.4 | 22.5 | 0.924 | manul |
| 11 | DMSO | PEEK | 26.7 | 25.8 | 26.3 | 0.897 | manul |
| 12 | DMSO | PEEK | 24.0 | 23.0 | 23.5 | 0.917 | manul |

Table 10-1: Complete table of the drop test results for Carbon/PEEK without treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Glycérol | PEEK | 67.8 | 3 | 1.88 |
| 2 | Formamide | PEEK | 54.5 | 3 | 1.29 |
| 3 | Diiodométhane | PEEK | 26.2 | 3 | 1.84 |
| 4 | DMSO | PEEK | 24.1 | 3 | 1.60 |

Table 10-2: Sum of initial drop test for Carbon/PEEK without treatment

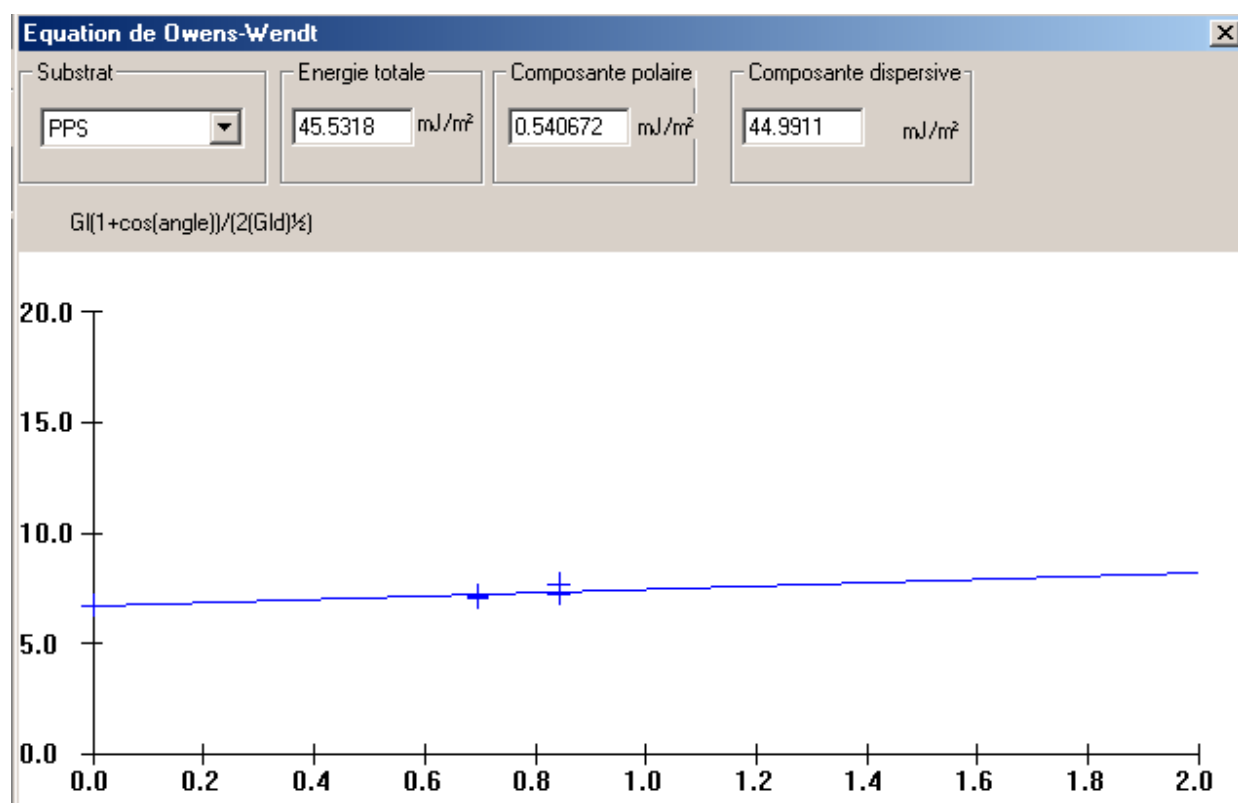


Figure 10-1: Drop test result of Owens-Wendt method for Carbon/PEEK without treatment

10.3.2 Carbon/PEEK – Corona treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | Cos | Mode |
|-----|-----------------|----------|-------|-------|--------|-------|---------|
| 1 | Glycérol | PEEK | 39.2 | 37.9 | 38.6 | 0.782 | m anual |
| 2 | Glycérol | PEEK | 31.0 | 30.6 | 30.8 | 0.859 | m anual |
| 3 | Glycérol | PEEK | 36.4 | 33.9 | 35.2 | 0.818 | m anual |
| 4 | Form amide | PEEK | 23.7 | 22.6 | 23.2 | 0.919 | m anual |
| 5 | Form amide | PEEK | 21.8 | 15.3 | 18.6 | 0.948 | m anual |
| 6 | Form amide | PEEK | 21.2 | 19.1 | 20.2 | 0.939 | m anual |
| 7 | D iiodom éthane | PEEK | 22.4 | 21.6 | 22.0 | 0.927 | m anual |
| 8 | D iiodom éthane | PEEK | 27.5 | 25.6 | 26.6 | 0.895 | m anual |
| 9 | D iiodom éthane | PEEK | 22.6 | 22.7 | 22.7 | 0.923 | m anual |
| 10 | D M S O | PEEK | 9.3 | 10.4 | 9.9 | 0.985 | m anual |
| 11 | D M S O | PEEK | 8.7 | 9.2 | 9.0 | 0.988 | m anual |
| 12 | D M S O | PEEK | 8.3 | 7.4 | 7.9 | 0.991 | m anual |

Table 10-3: Complete table of the drop test results for Carbon/PEEK with corona treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|-----------------|----------|--------|----------|-----------|
| 1 | Glycérol | PEEK | 34.8 | 3 | 3.17 |
| 2 | Form amide | PEEK | 20.6 | 3 | 1.91 |
| 3 | D iiodom éthane | PEEK | 23.7 | 3 | 2.01 |
| 4 | D M S O | PEEK | 8.9 | 3 | 0.82 |

Table 10-4: Sum of corona treatment for Carbon/PEEK

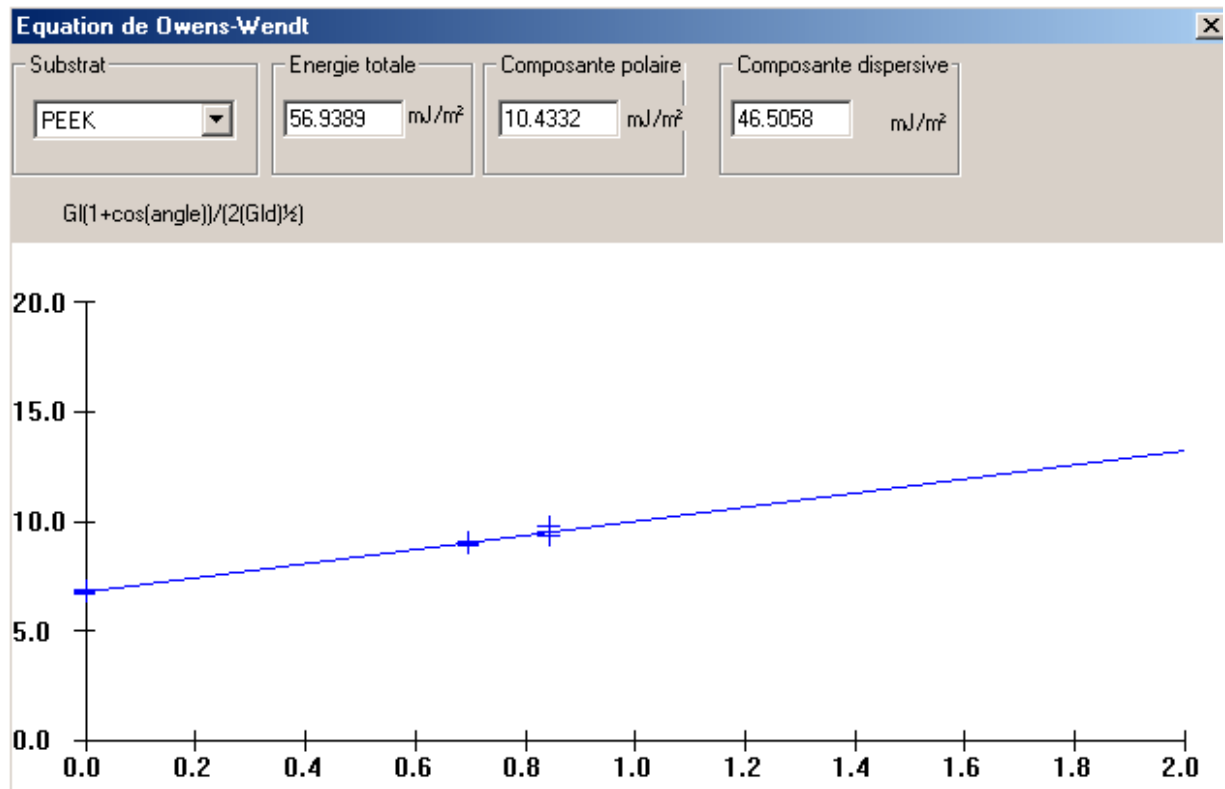


Figure 10-2: Drop test result of Owen-Wendt method for Carbon/PEEK with corona treatment

10.3.3 Carbon/PEEK – Abrasion treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | PEEK | 51.0 | 50.0 | 50.5 | 0.636 | marul |
| 2 | Glycérol | PEEK | 50.6 | 49.1 | 49.9 | 0.645 | marul |
| 3 | Glycérol | PEEK | 54.7 | 51.9 | 53.3 | 0.598 | marul |
| 4 | Formamide | PPEK | 44.6 | 42.0 | 43.3 | 0.728 | marul |
| 5 | Formamide | PPEK | 44.8 | 44.8 | 44.8 | 0.710 | marul |
| 6 | Formamide | PPEK | 46.5 | 43.9 | 45.2 | 0.705 | marul |
| 7 | Diiodométhane | PEEK | 16.1 | 16.6 | 16.4 | 0.960 | marul |
| 8 | Diiodométhane | PEEK | 11.0 | 11.8 | 11.4 | 0.980 | marul |
| 9 | Diiodométhane | PEEK | 11.6 | 12.8 | 12.2 | 0.977 | marul |
| 10 | DMSO | PEEK | 18.3 | 16.1 | 17.2 | 0.955 | marul |
| 11 | DMSO | PEEK | 16.8 | 16.6 | 16.7 | 0.958 | marul |
| 12 | DMSO | PEEK | 13.8 | 15.0 | 14.4 | 0.969 | marul |

Table 10-5: Complete table of the drop test results Carbon/PEEK with abrasion treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Glycérol | PEEK | 51.2 | 3 | 1.50 |
| 2 | Formamide | PPEK | 44.4 | 3 | 0.82 |
| 3 | Diiodométhane | PEEK | 13.3 | 3 | 2.17 |
| 4 | DMSO | PEEK | 16.1 | 3 | 1.22 |

Table 10-6: Sum of abrasion treatment for Carbon/PEEK

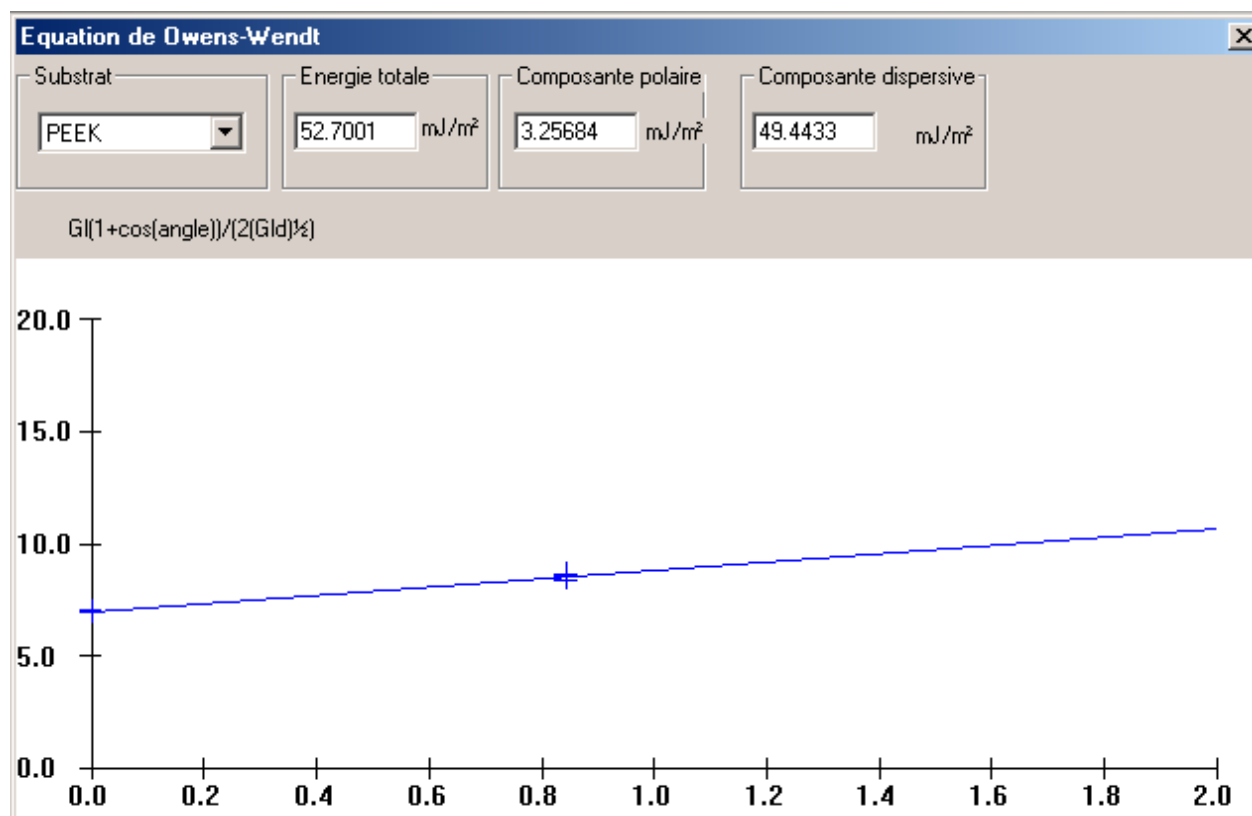


Figure 10-3: Drop test results of Owen-Wendt method for Carbon/PEEK with abrasion treatment

10.3.4 Carbon/PEEK – Flame treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | PEEK | 9.6 | 8.9 | 9.3 | 0.987 | manul |
| 2 | Glycérol | PEEK | 6.9 | 6.8 | 6.9 | 0.993 | manul |
| 3 | Glycérol | PEEK | 5.0 | 5.3 | 5.2 | 0.996 | manul |
| 4 | Formamide | PEEK | 7.6 | 7.4 | 7.5 | 0.991 | manul |
| 5 | Formamide | PEEK | 8.0 | 7.3 | 7.7 | 0.991 | manul |
| 6 | Formamide | PEEK | 5.6 | 6.6 | 6.1 | 0.994 | manul |
| 7 | Diiodométhane | PEEK | 17.4 | 18.5 | 18.0 | 0.951 | manul |
| 8 | Diiodométhane | PEEK | 20.0 | 19.8 | 19.9 | 0.940 | manul |
| 9 | Diiodométhane | PEEK | 24.3 | 25.7 | 25.0 | 0.906 | manul |
| 10 | DMSO | PEEK | 1.0 | 1.1 | 1.1 | 1.000 | manul |
| 11 | DMSO | PEEK | 2.2 | 2.3 | 2.3 | 0.999 | manul |
| 12 | DMSO | PEEK | 2.1 | 1.6 | 1.9 | 0.999 | manul |

Table 10-7: Complete table of the drop test results for Carbon/PEEK with flame treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Glycérol | PEEK | 7.1 | 3 | 1.68 |
| 2 | Formamide | PEEK | 7.1 | 3 | 0.70 |
| 3 | Diiodométhane | PEEK | 21.0 | 3 | 2.97 |
| 4 | DMSO | PEEK | 1.7 | 3 | 0.50 |

Table 10-8: Sum of flame treatment for Carbon/PEEK

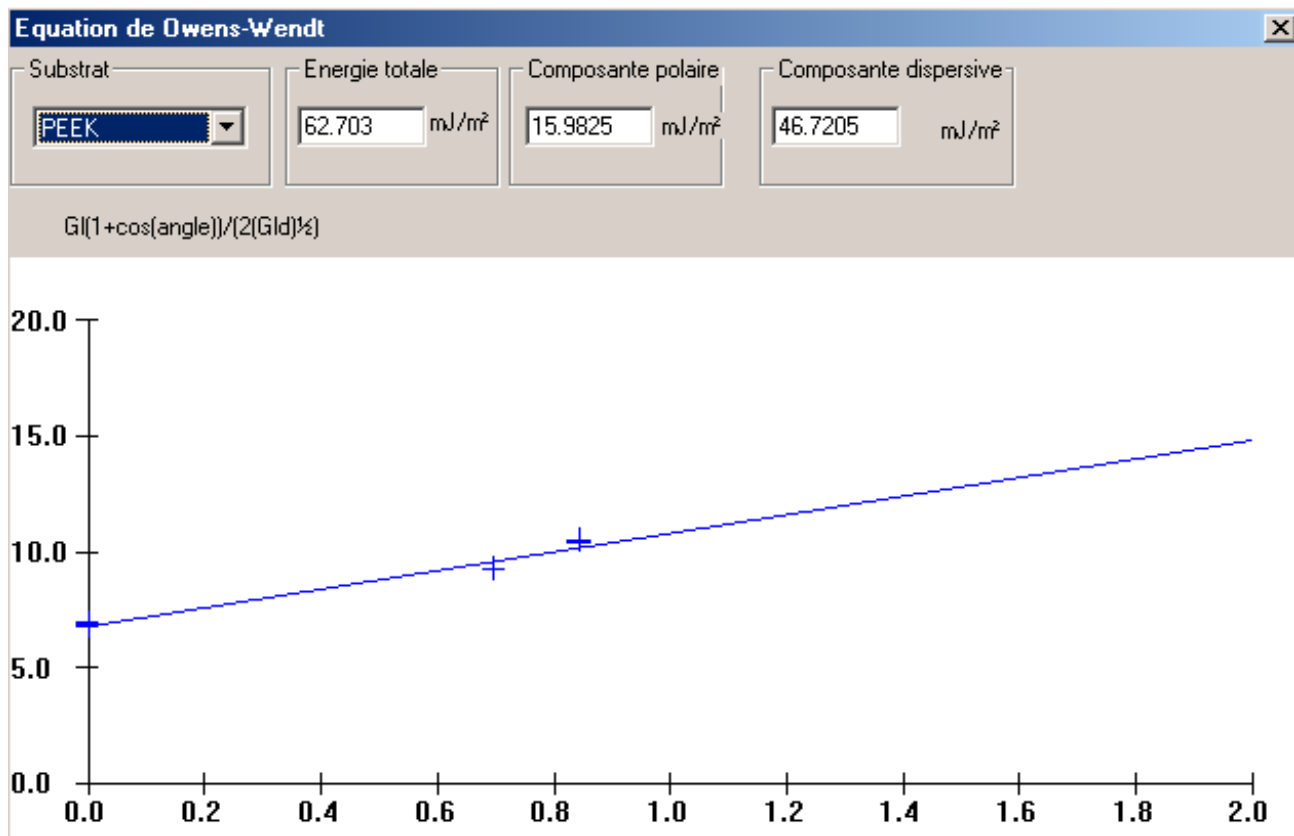


Figure 10-4: Drop test results of Owens-Wendt method for Carbon/PEEK with flame treatment

10.3.5 Carbon/PEEK – Plasma treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Formamide | PEEK | 10.0 | 10.1 | 10.1 | 0.985 | manul |
| 2 | Formamide | PEEK | 9.0 | 9.3 | 9.2 | 0.987 | manul |
| 3 | Formamide | PEEK | 11.9 | 11.5 | 11.7 | 0.979 | manul |
| 4 | Diiodométhane | PEEK | 26.2 | 26.7 | 26.5 | 0.895 | manul |
| 5 | Diiodométhane | PEEK | 23.6 | 23.7 | 23.7 | 0.916 | manul |
| 6 | Diiodométhane | PEEK | 24.7 | 25.8 | 25.3 | 0.904 | manul |
| 7 | DMSO | PEEK | 6.4 | 6.4 | 6.4 | 0.994 | manul |
| 8 | DMSO | PEEK | 7.4 | 6.6 | 7.0 | 0.993 | manul |
| 9 | DMSO | PEEK | 4.9 | 5.1 | 5.0 | 0.996 | manul |
| 10 | Glycérol | PEEK | 32.2 | 31.2 | 31.7 | 0.851 | manul |
| 11 | Glycérol | PEEK | 31.2 | 30.0 | 30.6 | 0.861 | manul |
| 12 | Glycérol | PEEK | 33.7 | 35.1 | 34.4 | 0.825 | manul |

Table 10-9: Complete table of the drop test results for Carbon/PEEK with plasma treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Formamide | PEEK | 10.3 | 3 | 1.06 |
| 2 | Diiodométhane | PEEK | 25.1 | 3 | 1.15 |
| 3 | DMSO | PEEK | 6.1 | 3 | 0.84 |
| 4 | Glycérol | PEEK | 32.2 | 3 | 1.60 |

Table 10-10: Sum of plasma treatment for Carbon/PEEK

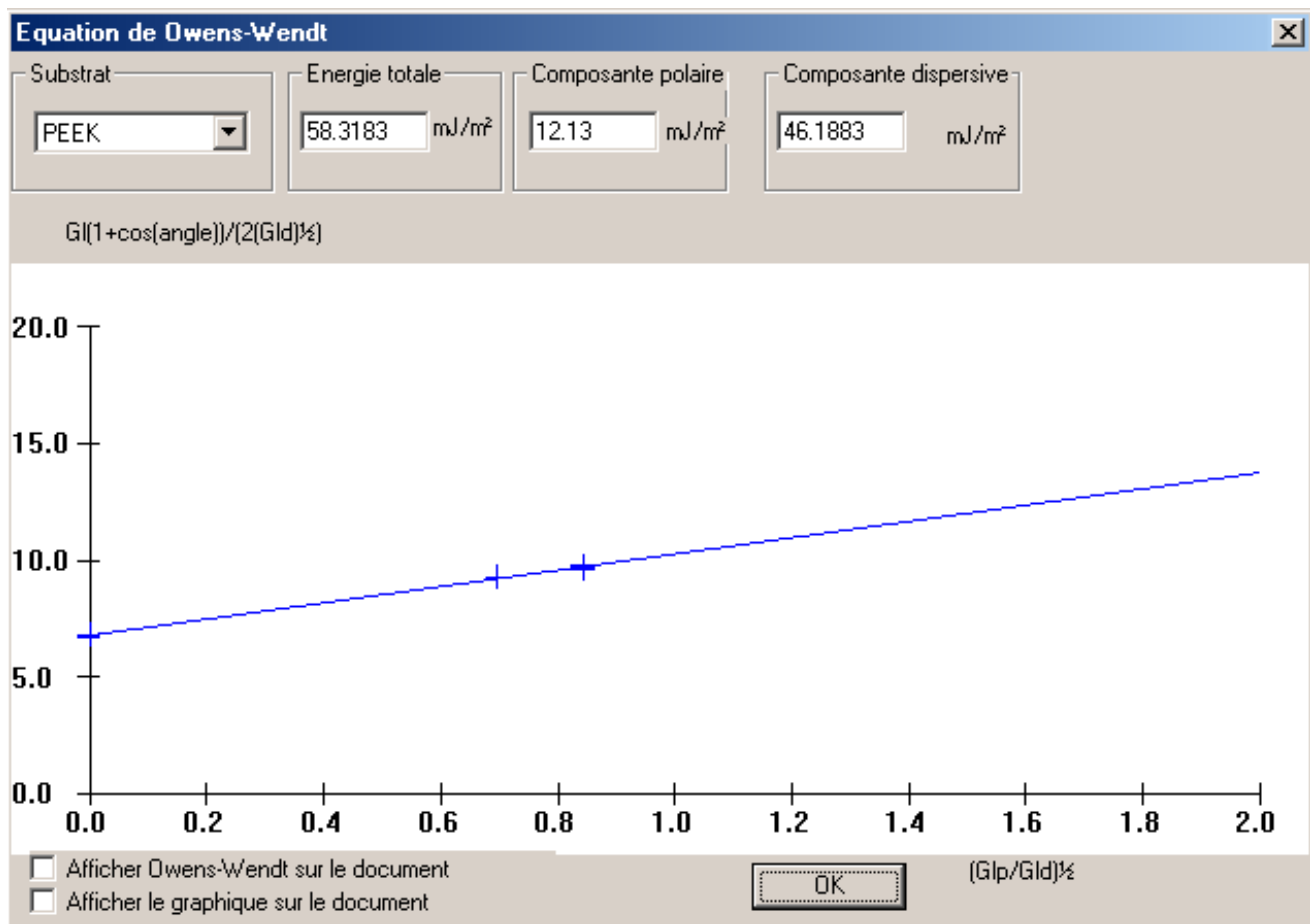


Figure 10-5: Drop test results of Owens-Wendt method for Carbon/PEEK with plasma treatment

10.3.6 Carbon/PEEK – Sandblasting treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | Cos | Mode |
|-----|---------------|----------|-------|-------|--------|-------|-------|
| 1 | Glycérol | PEEK | 52.3 | 53.0 | 52.7 | 0.607 | manul |
| 2 | Glycérol | PEEK | 49.5 | 47.7 | 48.6 | 0.661 | manul |
| 3 | Glycérol | PEEK | 52.2 | 49.5 | 50.9 | 0.631 | manul |
| 4 | Diiodométhane | PEEK | 7.8 | 7.3 | 7.6 | 0.991 | manul |
| 5 | Diiodométhane | PEEK | 8.7 | 8.3 | 8.5 | 0.989 | manul |
| 6 | Diiodométhane | PEEK | 11.2 | 11.0 | 11.1 | 0.981 | manul |
| 7 | D M S O | PEEK | 10.6 | 9.9 | 10.3 | 0.984 | manul |
| 8 | D M S O | PEEK | 10.3 | 9.2 | 9.8 | 0.986 | manul |
| 9 | D M S O | PEEK | 8.6 | 9.0 | 8.8 | 0.988 | manul |
| 10 | Formamide | PEEK | 47.7 | 43.9 | 45.8 | 0.697 | manul |
| 11 | Formamide | PEEK | 41.3 | 39.7 | 40.5 | 0.760 | manul |
| 12 | Formamide | PEEK | 42.2 | 40.9 | 41.6 | 0.748 | manul |

Table 10-11: Complete table of the drop test results for Carbon/PEEK with sandblasting treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|--------|----------|-----------|
| 1 | Glycérol | PEEK | 50.7 | 3 | 1.66 |
| 2 | Diiodométhane | PEEK | 9.0 | 3 | 1.50 |
| 3 | D M S O | PEEK | 9.6 | 3 | 0.60 |
| 4 | Formamide | PEEK | 42.6 | 3 | 2.29 |

Table 10-12: Sum of sandblasting treatment for Carbon/PEEK

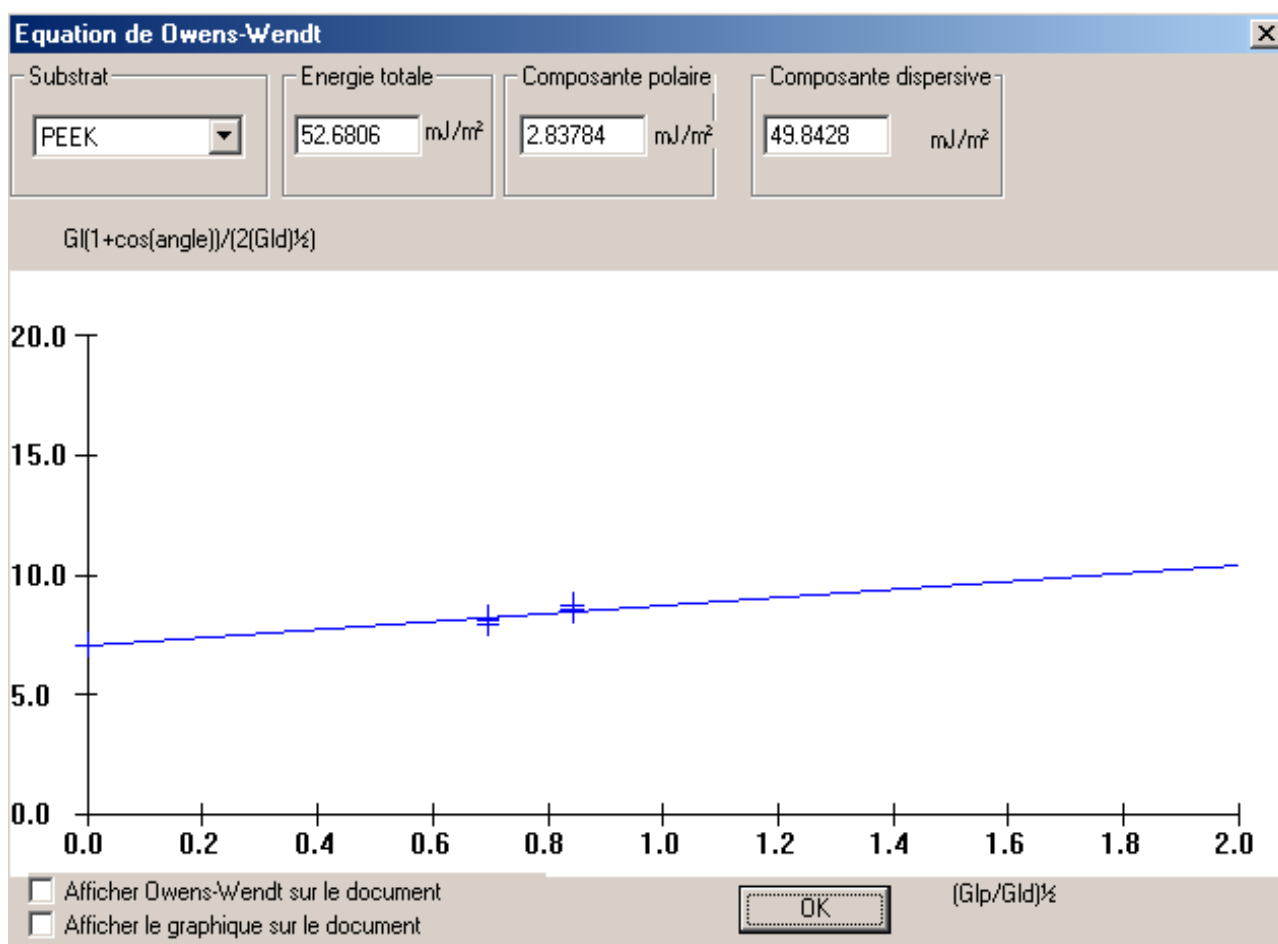


Figure 10-6: Drop test results of Owens-Wendt for Carbon/PEEK with sandblasting treatment

10.4 Appendix 4 – Carbon/PPS drop test results

10.4.1 Carbon/PPS – Without treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | Cos | Mode |
|-----|----------------|----------|-------|-------|--------|-------|---------|
| 1 | Glycérol | PPS | 67.8 | 68.4 | 68.1 | 0.373 | m anual |
| 2 | Glycérol | PPS | 69.2 | 65.2 | 67.2 | 0.388 | m anual |
| 3 | Glycérol | PPS | 63.8 | 61.7 | 62.8 | 0.458 | m anual |
| 4 | Form amide | PPS | 58.5 | 56.2 | 57.4 | 0.540 | m anual |
| 5 | Form amide | PPS | 60.6 | 57.0 | 58.8 | 0.518 | m anual |
| 6 | Form amide | PPS | 57.9 | 58.3 | 58.1 | 0.528 | m anual |
| 7 | Diiodom éthane | PPS | 28.6 | 26.7 | 27.7 | 0.886 | m anual |
| 8 | Diiodom éthane | PPS | 28.0 | 25.7 | 26.9 | 0.892 | m anual |
| 9 | Diiodom éthane | PPS | 29.9 | 26.6 | 28.3 | 0.881 | m anual |
| 10 | D M S O | PPS | 38.3 | 35.9 | 37.1 | 0.798 | m anual |
| 11 | D M S O | PPS | 37.2 | 34.3 | 35.8 | 0.812 | m anual |
| 12 | D M S O | PPS | 39.2 | 37.1 | 38.2 | 0.786 | m anual |

Table 10-13: Complete table of the drop test results for Carbon/PPS without treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|----------------|----------|--------|----------|-----------|
| 1 | Glycérol | PPS | 66.0 | 3 | 2.34 |
| 2 | Form amide | PPS | 58.1 | 3 | 0.59 |
| 3 | Diiodom éthane | PPS | 27.6 | 3 | 0.57 |
| 4 | D M S O | PPS | 37.0 | 3 | 0.98 |

Table 10-14: Sum of initial drop test for Carbon/PPS

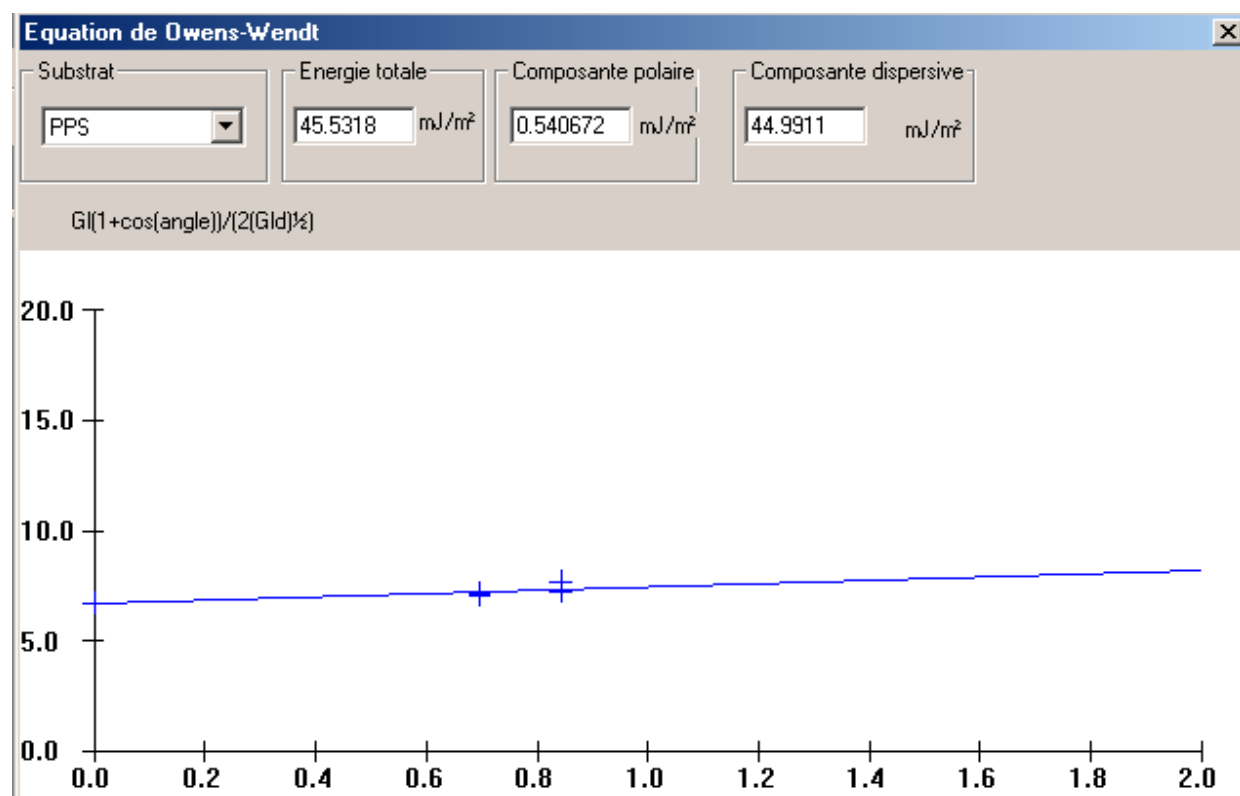


Figure 10-7: Drop test result of Owen-Wendt method for Carbon/PPS without treatment

10.4.2 Carbon/PPS – Corona treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | PPS | 41.7 | 38.4 | 40.1 | 0.765 | manul |
| 2 | Glycérol | PPS | 35.0 | 33.1 | 34.1 | 0.829 | manul |
| 3 | Glycérol | PPS | 39.6 | 42.1 | 40.9 | 0.756 | manul |
| 4 | Formamide | PPS | 27.0 | 30.2 | 28.6 | 0.878 | manul |
| 5 | Formamide | PPS | 35.5 | 33.2 | 34.4 | 0.826 | manul |
| 6 | Formamide | PPS | 40.2 | 38.2 | 39.2 | 0.775 | manul |
| 7 | Diiodométhane | PPS | 24.7 | 23.5 | 24.1 | 0.913 | manul |
| 8 | Diiodométhane | PPS | 25.5 | 23.7 | 24.6 | 0.909 | manul |
| 9 | Diiodométhane | PPS | 23.0 | 21.9 | 22.5 | 0.924 | manul |
| 10 | DMSO | PPS | 12.2 | 11.5 | 11.9 | 0.979 | manul |
| 11 | DMSO | PPS | 22.5 | 21.8 | 22.2 | 0.926 | manul |
| 12 | DMSO | PPS | 17.8 | 16.0 | 16.9 | 0.957 | manul |

Table 10-15: Complete table of the drop test results for Carbon/PPS with corona treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecartype |
|-----|---------------|----------|-------|----------|----------|
| 1 | Glycérol | PPS | 38.3 | 3 | 3.03 |
| 2 | Formamide | PPS | 34.0 | 3 | 4.33 |
| 3 | Diiodométhane | PPS | 23.7 | 3 | 0.92 |
| 4 | DMSO | PPS | 17.0 | 3 | 4.21 |

Table 10-16: Sum of corona treatment for Carbon/PPS

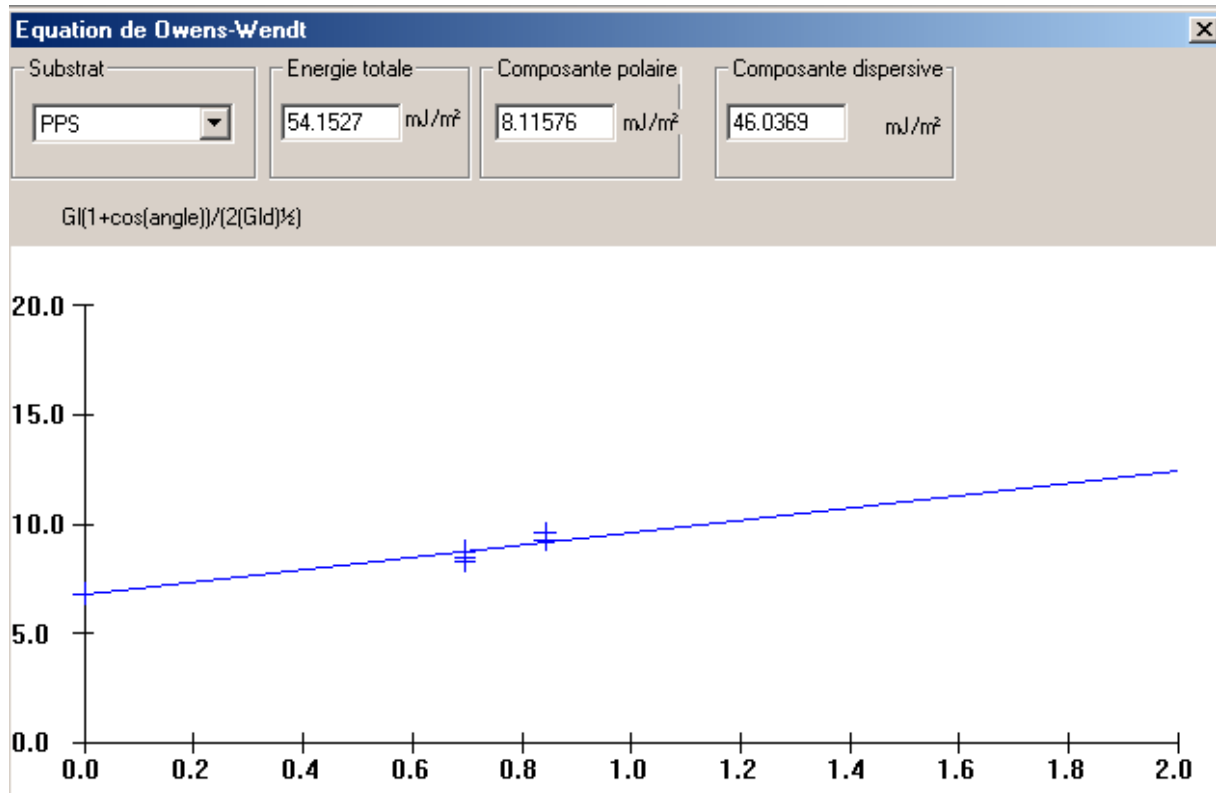


Figure 10-8: Drop test result of Owen-Wendt method for Carbon/PPS with corona treatment

10.4.3 Carbon/PPS – Abrasion treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | C os | M ode |
|-----|---------------|----------|-------|-------|--------|-------|-------|
| 1 | Glycérol | PPS | 60.8 | 59.4 | 60.1 | 0.498 | manul |
| 2 | Glycérol | PPS | 54.8 | 57.2 | 56.0 | 0.559 | manul |
| 3 | Glycérol | PPS | 62.6 | 59.2 | 60.9 | 0.486 | manul |
| 4 | Formamide | PPS | 59.5 | 55.6 | 57.6 | 0.537 | manul |
| 5 | Formamide | PPS | 56.0 | 55.0 | 55.5 | 0.566 | manul |
| 6 | Formamide | PPS | 57.3 | 53.0 | 55.2 | 0.571 | manul |
| 7 | Diiodométhane | PPS | 6.2 | 6.5 | 6.4 | 0.994 | manul |
| 8 | Diiodométhane | PPS | 5.8 | 5.6 | 5.7 | 0.995 | manul |
| 9 | Diiodométhane | PPS | 12.7 | 14.4 | 13.6 | 0.972 | manul |
| 10 | D M S O | PPS | 38.6 | 38.8 | 38.7 | 0.780 | manul |
| 11 | D M S O | PPS | 33.2 | 30.3 | 31.8 | 0.850 | manul |
| 12 | D M S O | PPS | 35.3 | 37.4 | 36.4 | 0.805 | manul |

Table 10-17: Complete table of drop test results for Carbon/PPS with abrasion treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|--------|----------|-----------|
| 1 | Glycérol | PPS | 59.0 | 3 | 2.15 |
| 2 | Formamide | PPS | 56.1 | 3 | 1.06 |
| 3 | Diiodométhane | PPS | 8.5 | 3 | 3.56 |
| 4 | D M S O | PPS | 35.6 | 3 | 2.89 |

Table 10-18: Sum of abrasion treatment for Carbon/PPS

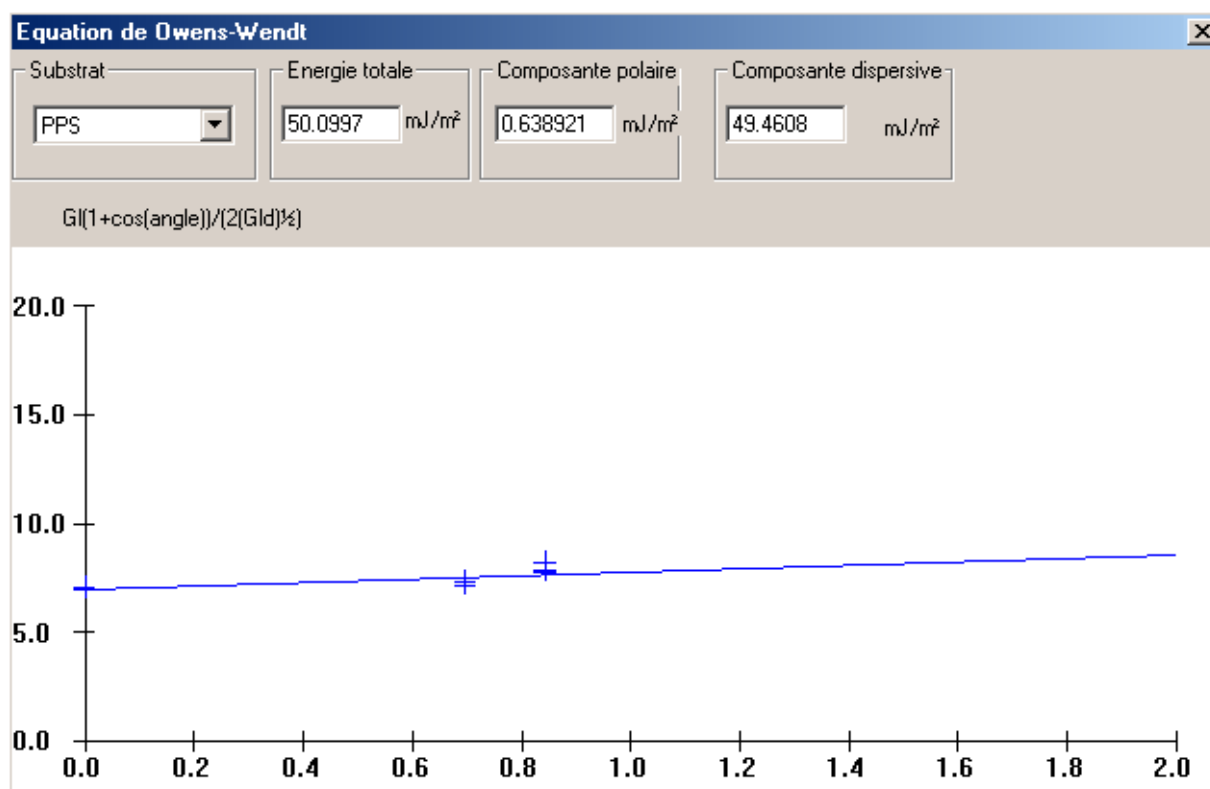


Figure 10-9: Drop test results by Owens-Wendt method for Carbon/PPS with abrasion treatment

10.4.4 Carbon/PPS – Flame treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | PPS | 17.8 | 15.4 | 16.6 | 0.958 | manul |
| 2 | Glycérol | PPS | 16.0 | 15.8 | 15.9 | 0.962 | manul |
| 3 | Glycérol | PPS | 12.4 | 12.2 | 12.3 | 0.977 | manul |
| 4 | Formamide | PPS | 17.5 | 17.0 | 17.3 | 0.955 | manul |
| 5 | Formamide | PPS | 25.7 | 25.0 | 25.4 | 0.904 | manul |
| 6 | Formamide | PPS | 26.3 | 26.0 | 26.2 | 0.898 | manul |
| 7 | Diiodométhane | PPS | 23.7 | 23.9 | 23.8 | 0.915 | manul |
| 8 | Diiodométhane | PPS | 23.4 | 24.7 | 24.1 | 0.913 | manul |
| 9 | Diiodométhane | PPS | 23.1 | 22.6 | 22.9 | 0.922 | manul |
| 10 | DMSO | PPS | 1.0 | 1.0 | 1.0 | 1.000 | manul |
| 11 | DMSO | PPS | 0.8 | 0.9 | 0.9 | 1.000 | manul |
| 12 | DMSO | PPS | 1.5 | 1.5 | 1.5 | 1.000 | manul |

Table 10-19: Complete table of drop test results for Carbon/PPS with flame treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Glycérol | PPS | 14.9 | 3 | 1.88 |
| 2 | Formamide | PPS | 22.9 | 3 | 4.02 |
| 3 | Diiodométhane | PPS | 23.6 | 3 | 0.52 |
| 4 | DMSO | PPS | 1.1 | 3 | 0.28 |

Table 10-20: Sum of flame treatment for Carbon/PPS

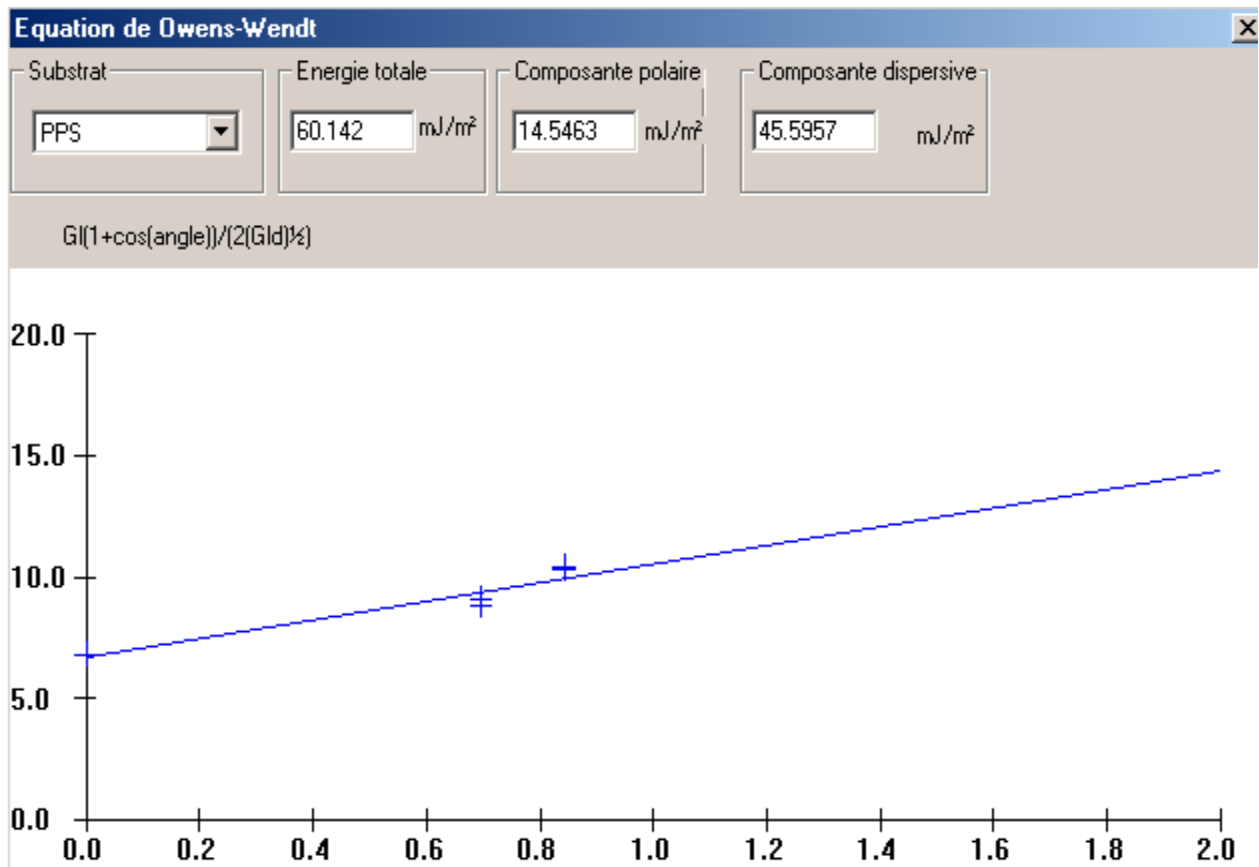


Figure 10-10: Drop test results by Owens-Wendt method for Carbon/PPS with flame treatment

10.4.5 Carbon/PPS – Plasma treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Formamide | PPS | 11.3 | 9.9 | 10.6 | 0.983 | manul |
| 2 | Formamide | PPS | 10.3 | 9.7 | 10.0 | 0.985 | manul |
| 3 | Formamide | PPS | 9.8 | 10.3 | 10.1 | 0.985 | manul |
| 4 | Diiodométhane | PPS | 25.2 | 24.9 | 25.1 | 0.906 | manul |
| 5 | Diiodométhane | PPS | 22.5 | 22.7 | 22.6 | 0.923 | manul |
| 6 | Diiodométhane | PPS | 22.7 | 24.2 | 23.5 | 0.917 | manul |
| 7 | DMSO | PPS | 2.3 | 2.1 | 2.2 | 0.999 | manul |
| 8 | DMSO | PPS | 1.7 | 1.8 | 1.8 | 1.000 | manul |
| 9 | DMSO | PPS | 1.9 | 2.2 | 2.1 | 0.999 | manul |
| 10 | Glycérol | PPS | 31.0 | 30.7 | 30.9 | 0.859 | manul |
| 11 | Glycérol | PPS | 29.9 | 29.9 | 29.9 | 0.867 | manul |
| 12 | Glycérol | PPS | 30.0 | 28.6 | 29.3 | 0.872 | manul |

Table 10-21: Complete table of drop test results for Carbon/PPS with plasma treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Formamide | PPS | 10.2 | 3 | 0.27 |
| 2 | Diiodométhane | PPS | 23.7 | 3 | 1.02 |
| 3 | DMSO | PPS | 2.0 | 3 | 0.19 |
| 4 | Glycérol | PPS | 30.0 | 3 | 0.64 |

Table 10-22: Sum of plasma treatment for Carbon/PPS

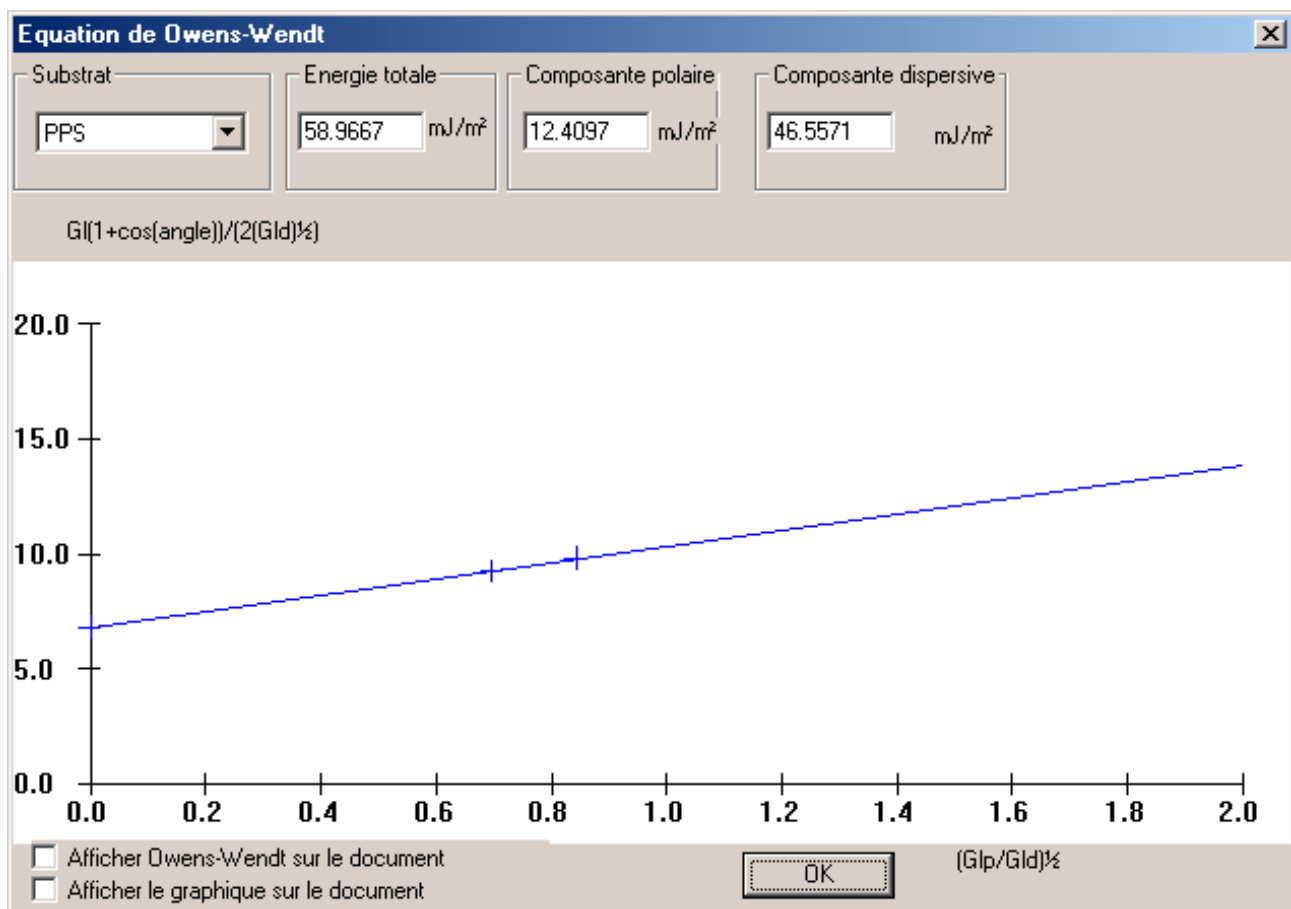


Figure 10-11: Drop test result by Owen-Wendt method for Carbon/PPS with plasma treatment

10.4.6 Carbon/PPS – Sandblasting treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | PPS | 65.9 | 67.6 | 66.8 | 0.395 | manul |
| 2 | Glycérol | PPS | 63.6 | 64.1 | 63.9 | 0.441 | manul |
| 3 | Glycérol | PPS | 64.1 | 65.2 | 64.7 | 0.428 | manul |
| 4 | Diiodométhane | PPS | 13.7 | 13.5 | 13.6 | 0.972 | manul |
| 5 | Diiodométhane | PPS | 9.5 | 9.6 | 9.6 | 0.986 | manul |
| 6 | Diiodométhane | PPS | 12.8 | 11.7 | 12.3 | 0.977 | manul |
| 7 | DMSO | PPS | 11.7 | 10.9 | 11.3 | 0.981 | manul |
| 8 | DMSO | PPS | 10.8 | 10.3 | 10.6 | 0.983 | manul |
| 9 | DMSO | PPS | 11.1 | 10.6 | 10.9 | 0.982 | manul |
| 10 | Formamide | PPS | 50.4 | 49.2 | 49.8 | 0.645 | manul |
| 11 | Formamide | PPS | 47.2 | 43.9 | 45.6 | 0.700 | manul |
| 12 | Formamide | PPS | 46.9 | 46.3 | 46.6 | 0.687 | manul |

Table 10-23: Complete table of drop test results for Carbon/PPS with sandblasting treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Glycérol | PPS | 65.1 | 3 | 1.22 |
| 2 | Diiodométhane | PPS | 11.8 | 3 | 1.68 |
| 3 | DMSO | PPS | 10.9 | 3 | 0.31 |
| 4 | Formamide | PPS | 47.3 | 3 | 1.81 |

Table 10-24: Sum of sandblasting treatment for Carbon/PPS

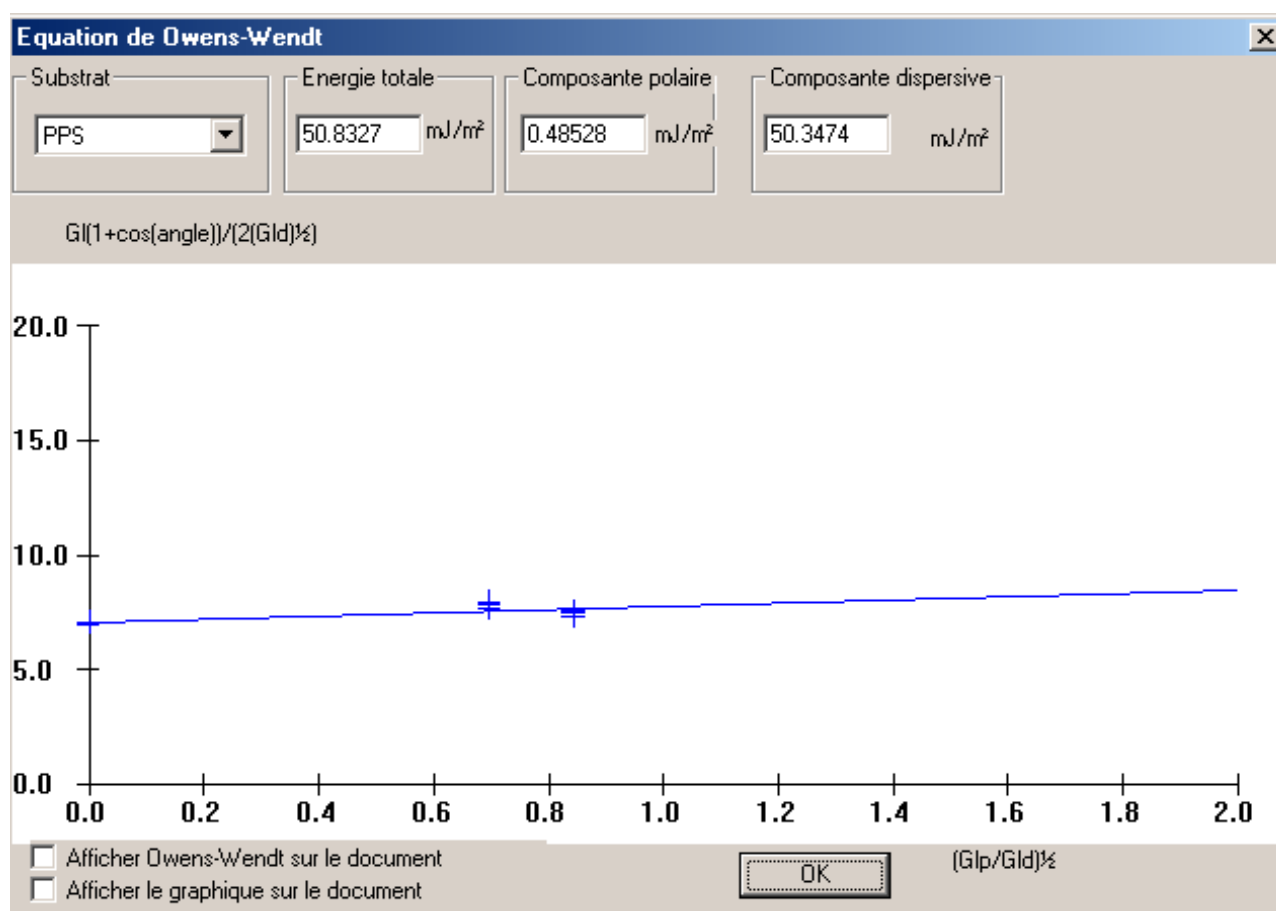


Figure 10-12: Drop test result by Owen-Wendt for Carbon/PPS with sandblasting treatment

10.5 Appendix 5 – Glass/Pa12 drop test results

10.5.1 Glass/Pa12 – Without treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | Cos | Mode |
|-----|----------------|----------|-------|-------|--------|-------|--------|
| 1 | Glycérol | Pa12 | 68.6 | 66.1 | 67.4 | 0.385 | m anul |
| 2 | Glycérol | Pa12 | 68.5 | 63.4 | 66.0 | 0.408 | m anul |
| 3 | Glycérol | Pa12 | 67.3 | 61.0 | 64.2 | 0.436 | m anul |
| 4 | Form amide | Pa12 | 61.5 | 56.2 | 58.9 | 0.517 | m anul |
| 5 | Form amide | Pa12 | 59.3 | 56.3 | 57.8 | 0.533 | m anul |
| 6 | Form amide | Pa12 | 62.1 | 57.5 | 59.8 | 0.503 | m anul |
| 7 | Diiodom éthane | Pa12 | 37.9 | 37.3 | 37.6 | 0.792 | m anul |
| 8 | Diiodom éthane | Pa12 | 43.1 | 40.8 | 42.0 | 0.744 | m anul |
| 9 | Diiodom éthane | Pa12 | 38.8 | 37.6 | 38.2 | 0.786 | m anul |
| 10 | D M S O | Pa12 | 46.9 | 44.5 | 45.7 | 0.698 | m anul |
| 11 | D M S O | Pa12 | 50.1 | 46.4 | 48.3 | 0.666 | m anul |
| 12 | D M S O | Pa12 | 51.8 | 47.4 | 49.6 | 0.648 | m anul |

Table 10-25: Complete table of the drop test results for Glass/Pa12 without treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|----------------|----------|--------|----------|-----------|
| 1 | Glycérol | Pa12 | 65.8 | 3 | 1.31 |
| 2 | Form amide | Pa12 | 58.8 | 3 | 0.82 |
| 3 | Diiodom éthane | Pa12 | 39.3 | 3 | 1.92 |
| 4 | D M S O | Pa12 | 47.9 | 3 | 1.62 |

Table 10-26: Sum of initial drop test for Glass/Pa12

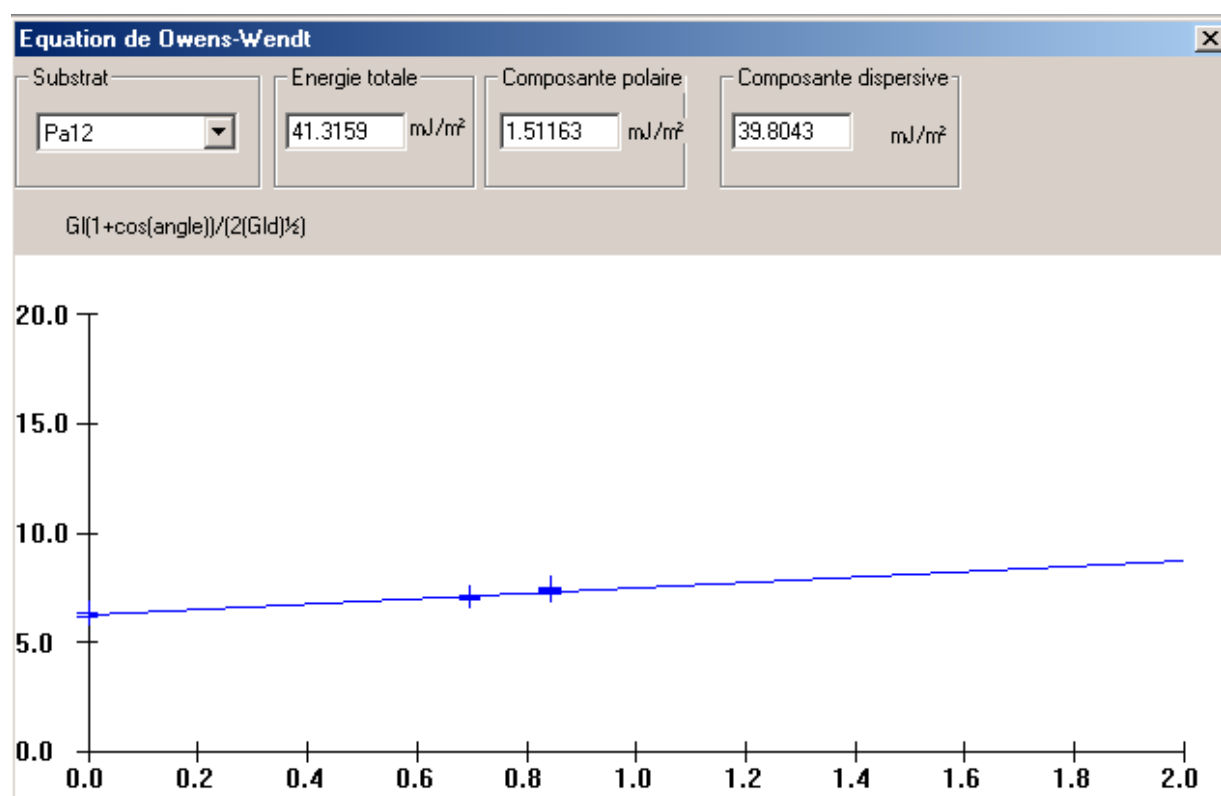


Figure 10-13: Drop test results by Owen-Wendt method for Glass/Pa12 without treatment

10.5.2 Glass/Pa12 – Corona treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | Pa12 | 65.6 | 61.9 | 63.8 | 0.442 | marul |
| 2 | Glycérol | Pa12 | 68.2 | 64.9 | 66.6 | 0.398 | marul |
| 3 | Glycérol | Pa12 | 57.1 | 56.9 | 57.0 | 0.545 | marul |
| 4 | Formamide | Pa12 | 47.3 | 40.8 | 44.1 | 0.719 | marul |
| 5 | Formamide | Pa12 | 43.9 | 42.6 | 43.3 | 0.728 | marul |
| 6 | Formamide | Pa12 | 53.0 | 50.1 | 51.6 | 0.622 | marul |
| 7 | Diiodométhane | Pa12 | 34.2 | 33.3 | 33.8 | 0.831 | marul |
| 8 | Diiodométhane | Pa12 | 33.1 | 28.8 | 31.0 | 0.858 | marul |
| 9 | Diiodométhane | Pa12 | 32.4 | 31.0 | 31.7 | 0.851 | marul |
| 10 | DMSO | Pa12 | 33.3 | 28.4 | 30.9 | 0.859 | marul |
| 11 | DMSO | Pa12 | 34.9 | 33.0 | 34.0 | 0.830 | marul |
| 12 | DMSO | Pa12 | 34.7 | 30.7 | 32.7 | 0.842 | marul |

Table 10-27: Complete table of drop test results for Glass/Pa12 with corona treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Glycérol | Pa12 | 62.4 | 3 | 4.01 |
| 2 | Formamide | Pa12 | 46.3 | 3 | 3.74 |
| 3 | Diiodométhane | Pa12 | 32.1 | 3 | 1.18 |
| 4 | DMSO | Pa12 | 32.5 | 3 | 1.27 |

Table 10-28: Sum of corona treatment for Glass/Pa12

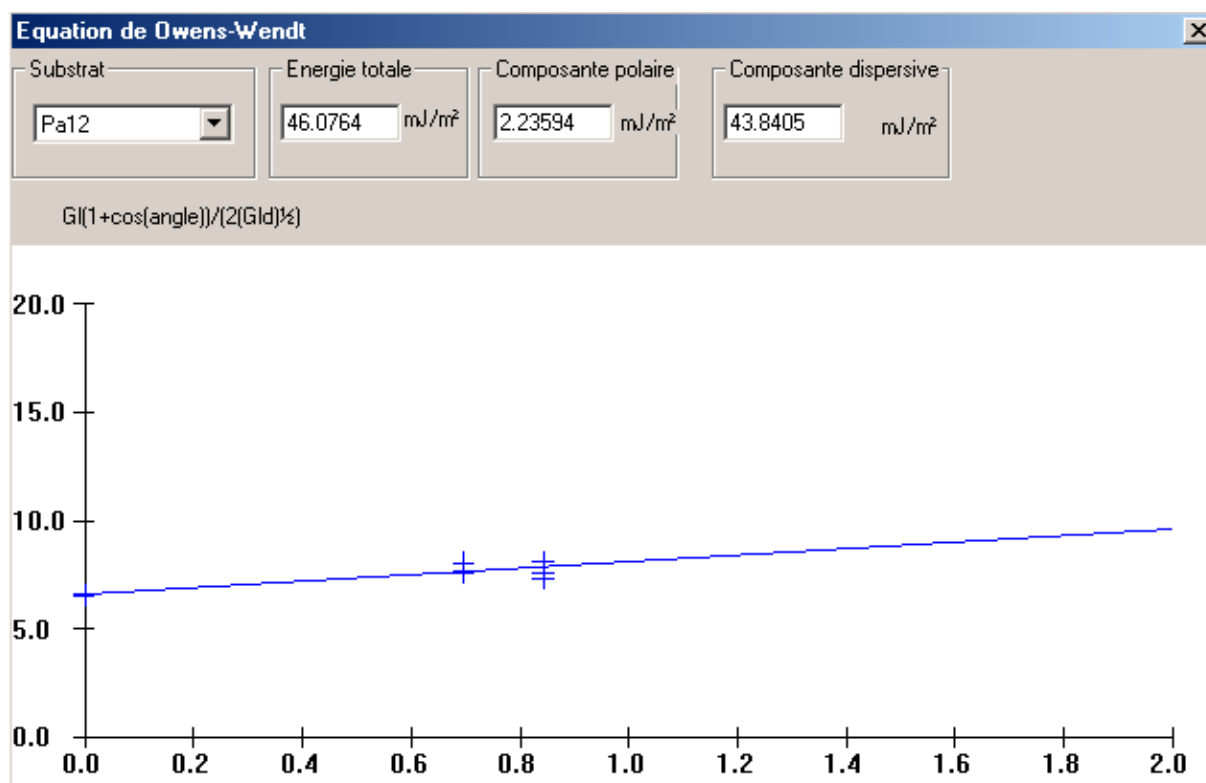


Figure 10-14: Drop test results by Owen-Wendt method for Glass/Pa12 with corona treatment

10.5.3 Glass/Pa12 – Abrasion treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|--------|
| 1 | Glycérol | Pal2 | 66.3 | 60.6 | 63.5 | 0.447 | manuel |
| 2 | Glycérol | Pal2 | 64.9 | 63.8 | 64.4 | 0.433 | manuel |
| 3 | Glycérol | Pal2 | 65.9 | 66.0 | 66.0 | 0.408 | manuel |
| 4 | Formamide | Pal2 | 59.4 | 58.0 | 58.7 | 0.520 | manuel |
| 5 | Formamide | Pal2 | 58.4 | 57.7 | 58.1 | 0.529 | manuel |
| 6 | Formamide | Pal2 | 54.4 | 51.8 | 53.1 | 0.600 | manuel |
| 7 | Diiodométhane | Pal2 | 24.8 | 25.3 | 25.1 | 0.906 | manuel |
| 8 | Diiodométhane | Pal2 | 26.9 | 26.4 | 26.7 | 0.894 | manuel |
| 9 | Diiodométhane | Pal2 | 32.3 | 31.7 | 32.0 | 0.848 | manuel |
| 10 | DMSO | Pal2 | 28.5 | 30.3 | 29.4 | 0.871 | manuel |
| 11 | DMSO | Pal2 | 23.2 | 24.8 | 24.0 | 0.914 | manuel |
| 12 | DMSO | Pal2 | 29.5 | 30.6 | 30.1 | 0.866 | manuel |

Table 10-29: Complete table of drop test results for Glass/Pa12 with abrasion treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Glycérol | Pal2 | 64.6 | 3 | 1.03 |
| 2 | Formamide | Pal2 | 56.6 | 3 | 2.50 |
| 3 | Diiodométhane | Pal2 | 27.9 | 3 | 2.97 |
| 4 | DMSO | Pal2 | 27.8 | 3 | 2.71 |

Table 10-30: Sum of abrasion treatment for Glass/Pa12

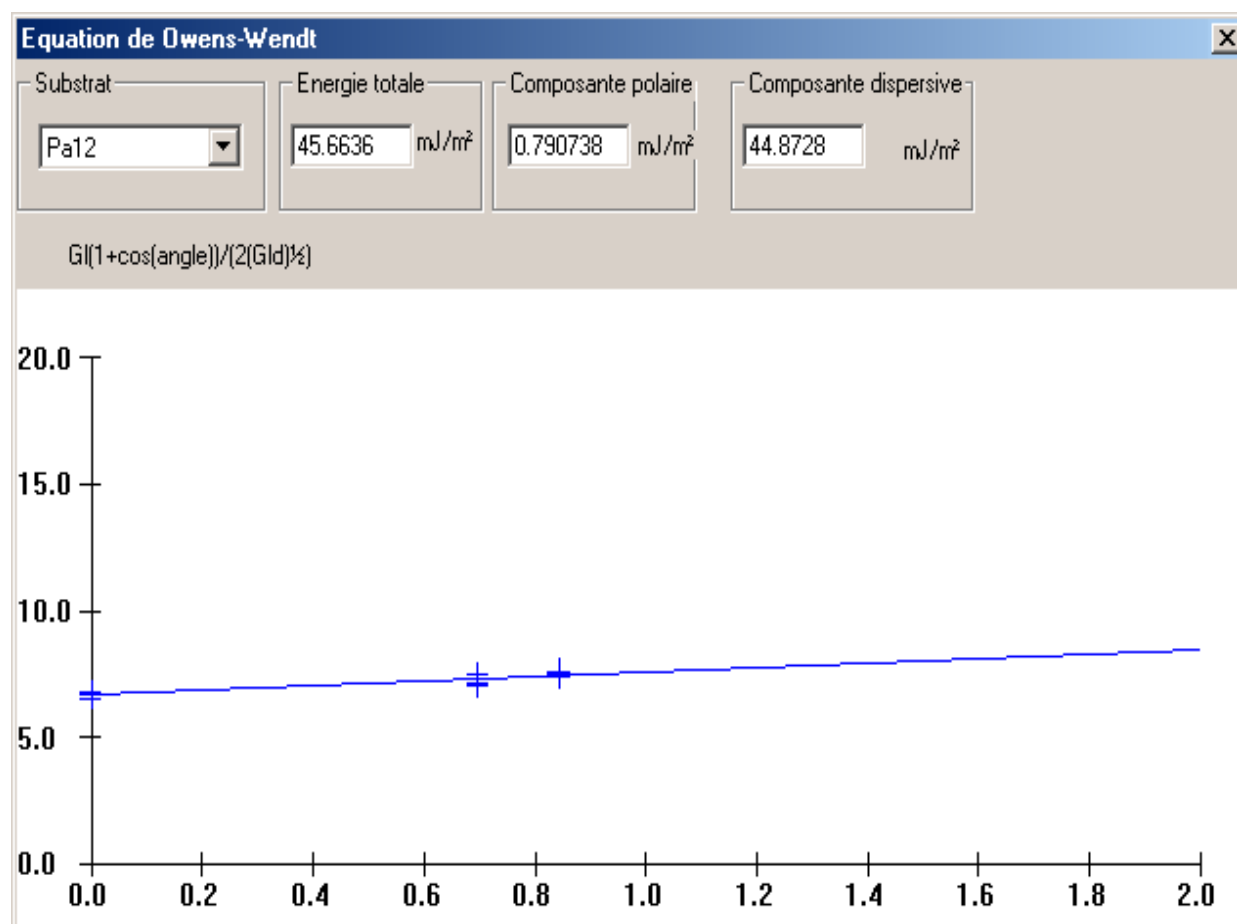


Figure 10-15: Drop test results by Owen-Wendt method for Glass/Pa12 with abrasion treatment

10.5.4 Glass/Pa12 – Flame treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | Cos | Mode |
|-----|---------------|----------|-------|-------|--------|-------|-------|
| 1 | Glycérol | PA12 | 45.5 | 44.7 | 45.1 | 0.706 | manul |
| 2 | Glycérol | PA12 | 36.5 | 37.1 | 36.8 | 0.801 | manul |
| 3 | Glycérol | PA12 | 42.7 | 42.8 | 42.8 | 0.734 | manul |
| 4 | Formamide | PA12 | 37.7 | 35.7 | 36.7 | 0.802 | manul |
| 5 | Formamide | PA12 | 16.7 | 16.4 | 16.6 | 0.959 | manul |
| 6 | Formamide | PA12 | 15.8 | 16.7 | 16.3 | 0.960 | manul |
| 7 | Diiodométhane | PA12 | 30.0 | 30.7 | 30.4 | 0.863 | manul |
| 8 | Diiodométhane | PA12 | 32.2 | 32.2 | 32.2 | 0.846 | manul |
| 9 | Diiodométhane | PA12 | 25.2 | 25.4 | 25.3 | 0.904 | manul |
| 10 | DMSO | PA12 | 2.2 | 2.2 | 2.2 | 0.999 | manul |
| 11 | DMSO | PA12 | 12.6 | 14.0 | 13.3 | 0.973 | manul |
| 12 | DMSO | PA12 | 12.1 | 12.7 | 12.4 | 0.977 | manul |

Table 10-31: Complete table of drop test results for Glass/Pa12 with flame treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|--------|----------|-----------|
| 1 | Glycérol | PA12 | 41.5 | 3 | 3.49 |
| 2 | Formamide | PA12 | 23.2 | 3 | 9.57 |
| 3 | Diiodométhane | PA12 | 29.3 | 3 | 2.92 |
| 4 | DMSO | PA12 | 9.3 | 3 | 5.03 |

Table 10-32: Sum of flame treatment for Glass/Pa12

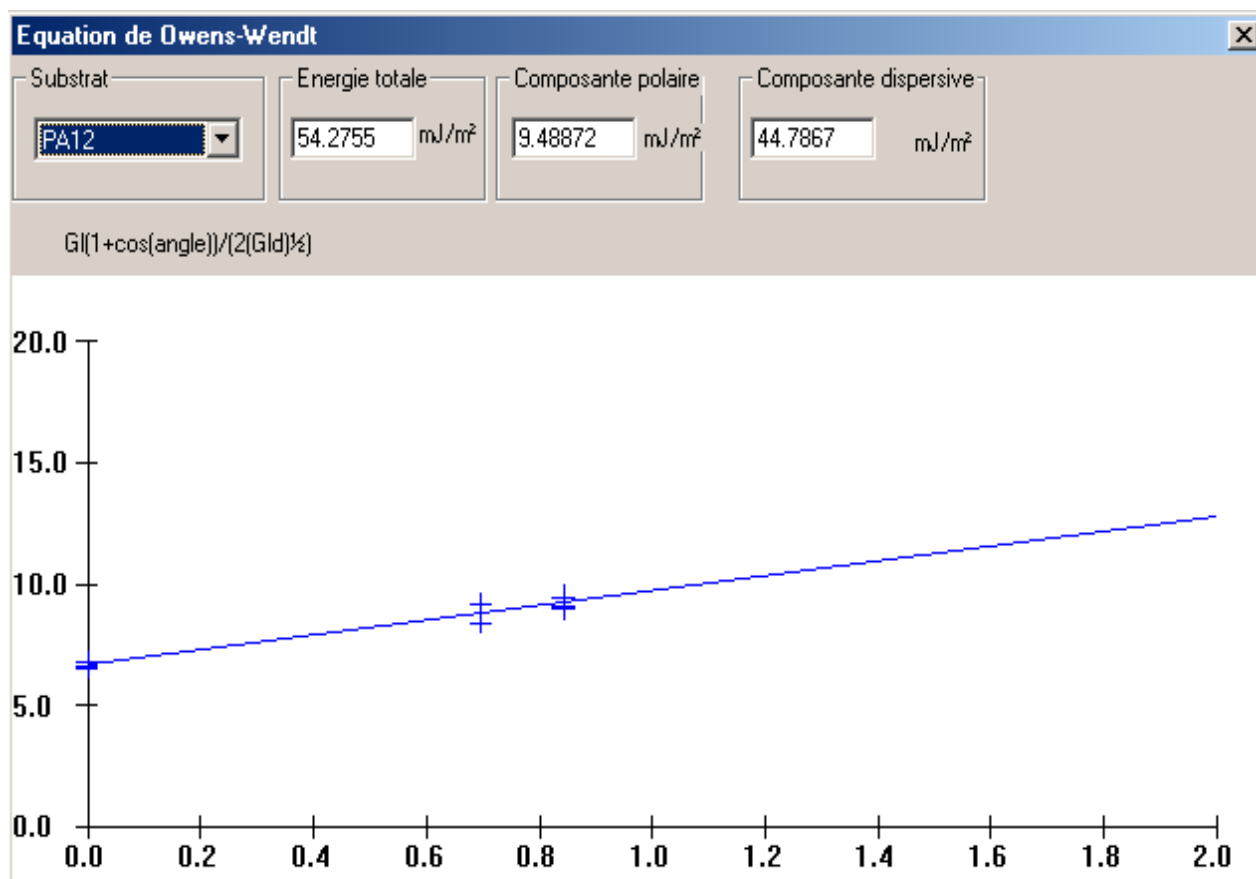


Figure 10-16: Drop test results by Owens-Wendt method for Glass/Pa12 with flame treatment

10.5.5 Glass/Pa12 – Plasma treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Formamide | PA12 | 10.8 | 11.6 | 11.2 | 0.981 | manul |
| 2 | Formamide | PA12 | 13.6 | 13.8 | 13.7 | 0.972 | manul |
| 3 | Formamide | PA12 | 10.1 | 9.0 | 9.6 | 0.986 | manul |
| 4 | Diiodométhane | PA12 | 24.0 | 22.0 | 23.0 | 0.921 | manul |
| 5 | Diiodométhane | PA12 | 21.5 | 19.8 | 20.7 | 0.936 | manul |
| 6 | Diiodométhane | PA12 | 22.1 | 22.0 | 22.1 | 0.927 | manul |
| 7 | DMSO | PA12 | 1.6 | 1.5 | 1.6 | 1.000 | manul |
| 8 | DMSO | PA12 | 1.9 | 1.8 | 1.9 | 0.999 | manul |
| 9 | DMSO | PA12 | 4.1 | 5.9 | 5.0 | 0.996 | manul |
| 10 | Glycérol | PA12 | 25.8 | 24.0 | 24.9 | 0.907 | manul |
| 11 | Glycérol | PA12 | 30.6 | 29.2 | 29.9 | 0.867 | manul |
| 12 | Glycérol | PA12 | 27.3 | 26.6 | 27.0 | 0.891 | manul |

Table 10-33: Complete table of drop test results for Glass/Pa12 with plasma treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Formamide | PA12 | 11.5 | 3 | 1.71 |
| 2 | Diiodométhane | PA12 | 21.9 | 3 | 0.97 |
| 3 | DMSO | PA12 | 2.8 | 3 | 1.56 |
| 4 | Glycérol | PA12 | 27.3 | 3 | 2.05 |

Table 10-34: Sum of plasma treatment Glass/Pa12

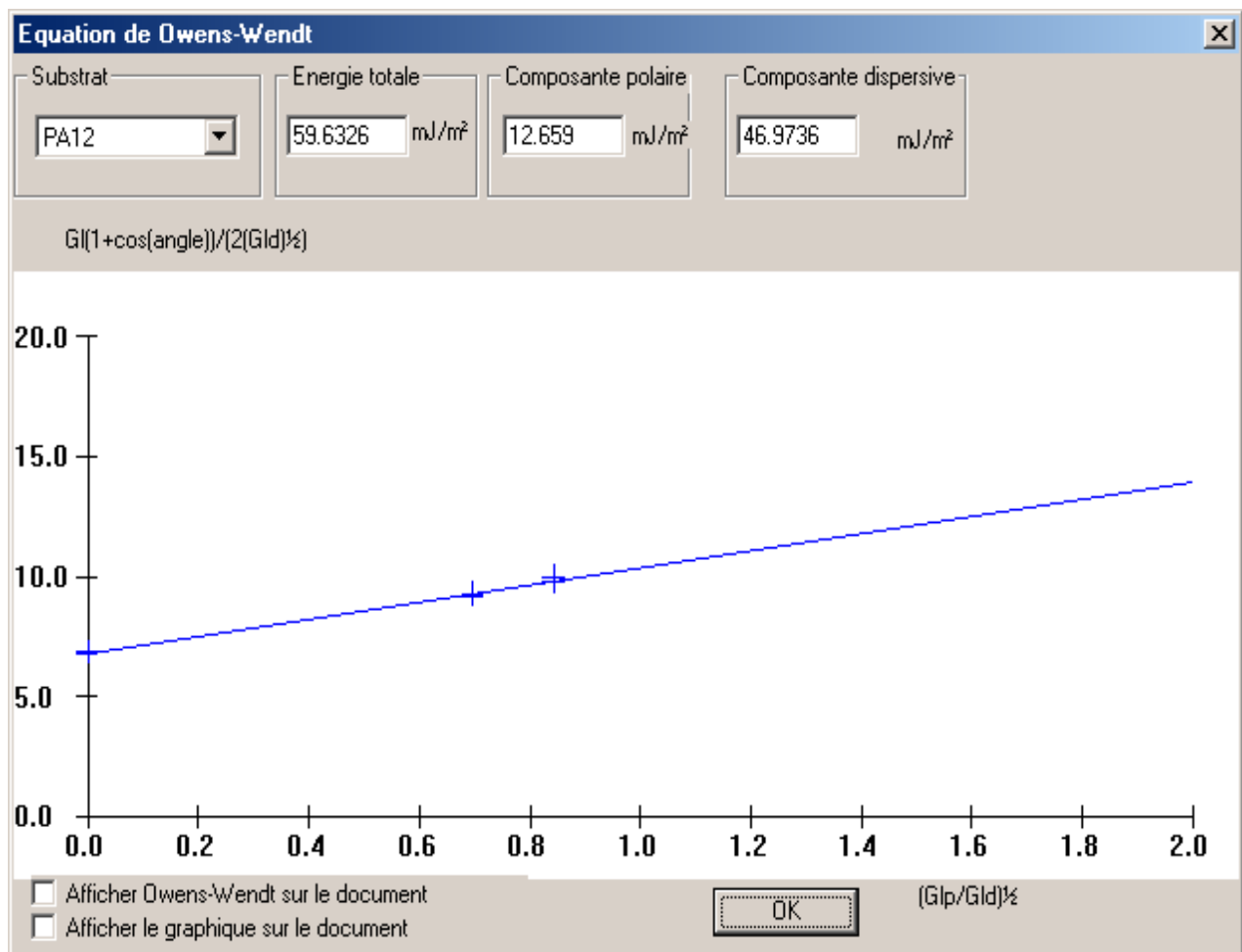


Figure 10-17: Drop test results by Owens-Wendt method for Glass/PA12 with plasma treatment

10.5.6 Pa12 – Sandblasting treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | C os | M ode |
|-----|---------------|----------|-------|-------|--------|-------|-------|
| 1 | Glycérol | PA12 | 48.5 | 50.6 | 49.6 | 0.649 | manul |
| 2 | Glycérol | PA12 | 51.6 | 53.6 | 52.6 | 0.607 | manul |
| 3 | Glycérol | PA12 | 58.2 | 56.7 | 57.5 | 0.538 | manul |
| 4 | Diiodométhane | PA12 | 27.3 | 27.9 | 27.6 | 0.886 | manul |
| 5 | Diiodométhane | PA12 | 17.7 | 15.8 | 16.8 | 0.958 | manul |
| 6 | Diiodométhane | PA12 | 15.6 | 15.8 | 15.7 | 0.963 | manul |
| 7 | DMSO | PA12 | 10.7 | 10.0 | 10.4 | 0.984 | manul |
| 8 | DMSO | PA12 | 11.6 | 12.1 | 11.9 | 0.979 | manul |
| 9 | DMSO | PA12 | 8.1 | 9.4 | 8.8 | 0.988 | manul |
| 10 | Formamide | PA12 | 39.0 | 38.8 | 38.9 | 0.778 | manul |
| 11 | Formamide | PA12 | 40.7 | 40.2 | 40.5 | 0.761 | manul |
| 12 | Formamide | PA12 | 38.9 | 38.0 | 38.5 | 0.783 | manul |

Table 10-35: Complete table of drop test results for Glass/Pa12 with sandblasting treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|--------|----------|-----------|
| 1 | Glycérol | PA12 | 53.2 | 3 | 3.25 |
| 2 | Diiodométhane | PA12 | 20.0 | 3 | 5.38 |
| 3 | DMSO | PA12 | 10.3 | 3 | 1.27 |
| 4 | Formamide | PA12 | 39.3 | 3 | 0.86 |

Table 10-36: Sum of sandblasting treatment for Glass/Pa12

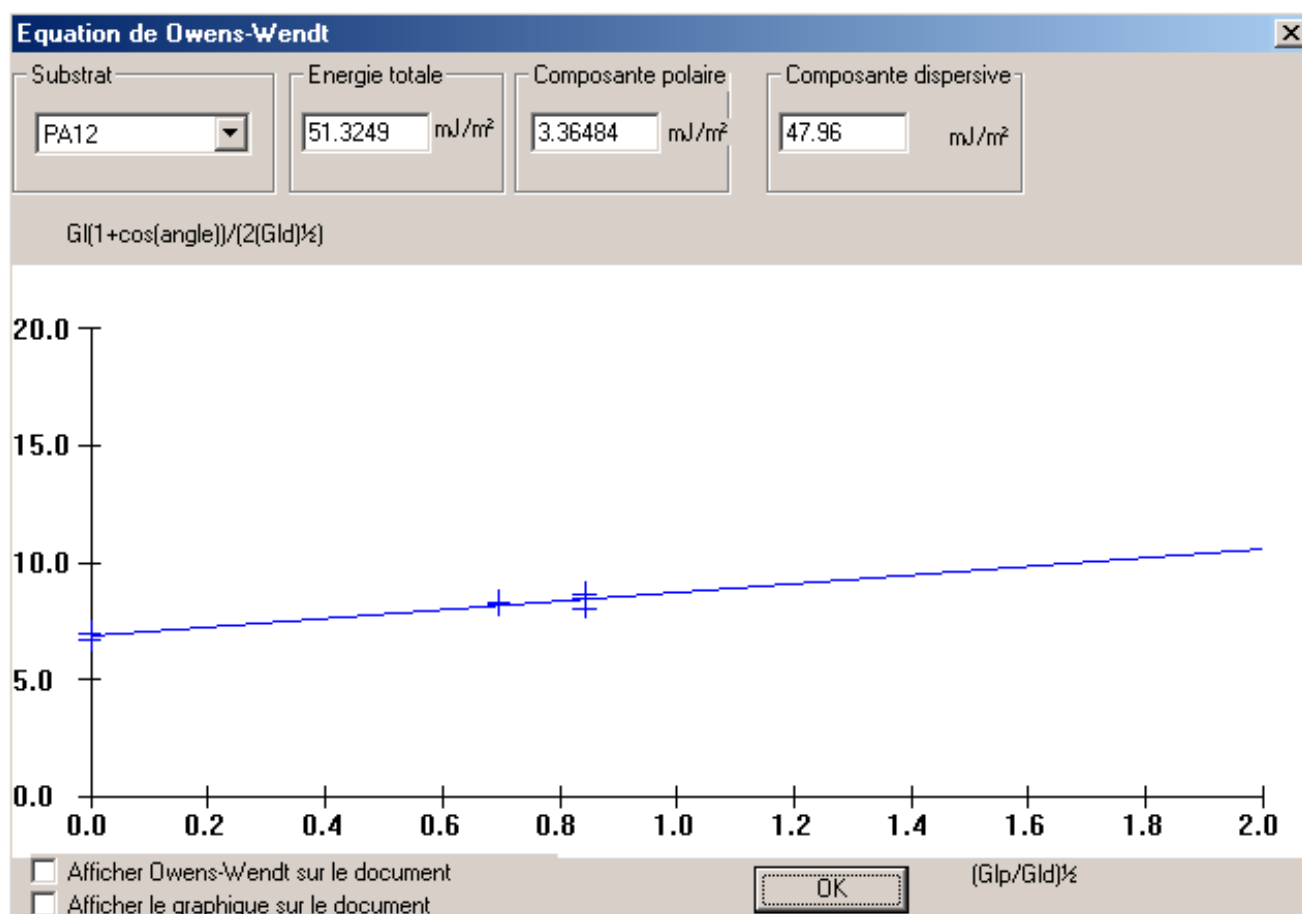


Figure 10-18: Drop test results by Owens-Wendt method for Glass/Pa12 with plasma treatment

10.6 Appendix 6 – Aluminum 2017 drop test results

10.6.1 Aluminum 2017– Without treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|-----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | Aluminium | 83.2 | 80.2 | 81.7 | 0.144 | manul |
| 2 | Glycérol | Aluminium | 80.8 | 73.4 | 77.1 | 0.223 | manul |
| 3 | Glycérol | Aluminium | 78.8 | 75.2 | 77.0 | 0.225 | manul |
| 4 | Formamide | Aluminium | 57.6 | 54.8 | 56.2 | 0.556 | manul |
| 5 | Formamide | Aluminium | 57.6 | 55.8 | 56.7 | 0.549 | manul |
| 6 | Formamide | Aluminium | 56.9 | 55.4 | 56.2 | 0.557 | manul |
| 7 | Diiodométhane | Aluminium | 34.7 | 33.3 | 34.0 | 0.829 | manul |
| 8 | Diiodométhane | Aluminium | 36.5 | 33.9 | 35.2 | 0.817 | manul |
| 9 | Diiodométhane | Aluminium | 37.0 | 39.3 | 38.2 | 0.786 | manul |
| 10 | DMSO | Aluminium | 51.0 | 50.2 | 50.6 | 0.635 | manul |
| 11 | DMSO | Aluminium | 54.1 | 51.7 | 52.9 | 0.603 | manul |
| 12 | DMSO | Aluminium | 56.8 | 53.1 | 55.0 | 0.574 | manul |

Table 10-37: Complete table of the drop test results for Aluminum 2017 without treatment

| N° | Liquide | Substrat | Moyen | Nb. Mes. | Ecart type |
|----|---------------|-----------|-------|----------|------------|
| 1 | Glycérol | Aluminium | 78.6 | 3 | 2.19 |
| 2 | Formamide | Aluminium | 56.4 | 3 | 0.25 |
| 3 | Diiodométhane | Aluminium | 35.8 | 3 | 1.74 |
| 4 | DMSO | Aluminium | 52.8 | 3 | 1.78 |

Table 10-38: Sum of initial drop test for Aluminum 2017

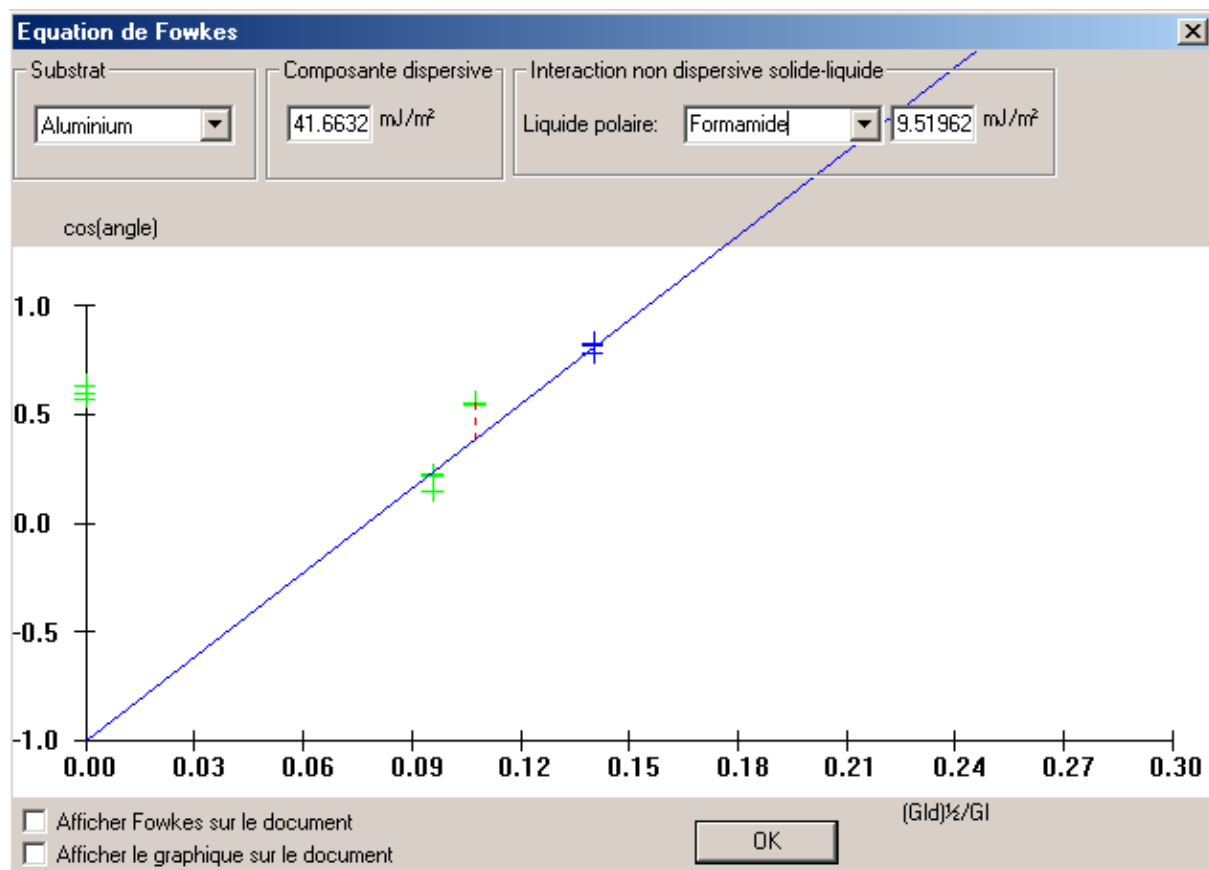


Figure 10-19: Drop test results by Fowkes method for Aluminum 2017 without treatment

10.6.2 Aluminum 2017 – Abrasion (80) treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | C os | M ode |
|-----|-----------------|-----------|-------|-------|--------|-------|---------|
| 1 | Glycérol | Aluminium | 57.0 | 53.0 | 55.0 | 0.574 | m anual |
| 2 | Glycérol | Aluminium | 49.3 | 50.5 | 49.9 | 0.644 | m anual |
| 3 | Glycérol | Aluminium | 55.5 | 57.7 | 56.6 | 0.550 | m anual |
| 4 | Form amide | Aluminium | 41.4 | 39.0 | 40.2 | 0.764 | m anual |
| 5 | Form amide | Aluminium | 38.7 | 44.6 | 41.7 | 0.747 | m anual |
| 6 | Form amide | Aluminium | 46.4 | 44.8 | 45.6 | 0.700 | m anual |
| 7 | D iiodom éthane | Aluminium | 36.8 | 34.1 | 35.5 | 0.815 | m anual |
| 8 | D iiodom éthane | Aluminium | 38.3 | 36.5 | 37.4 | 0.794 | m anual |
| 9 | D iiodom éthane | Aluminium | 40.0 | 40.9 | 40.5 | 0.761 | m anual |
| 10 | D M S O | Aluminium | 27.6 | 26.8 | 27.2 | 0.889 | m anual |
| 11 | D M S O | Aluminium | 30.7 | 28.0 | 29.4 | 0.872 | m anual |
| 12 | D M S O | Aluminium | 39.2 | 39.0 | 39.1 | 0.776 | m anual |

Table 10-39: Complete table of drop test results for Aluminum 2017 with abrasion (80) treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|-----------------|-----------|--------|----------|-----------|
| 1 | Glycérol | Aluminium | 53.8 | 3 | 2.86 |
| 2 | Form amide | Aluminium | 42.5 | 3 | 2.28 |
| 3 | D iiodom éthane | Aluminium | 37.8 | 3 | 2.06 |
| 4 | D M S O | Aluminium | 31.9 | 3 | 5.18 |

Table 10-40: Sum of abrasion (80) treatment for Aluminum 2017

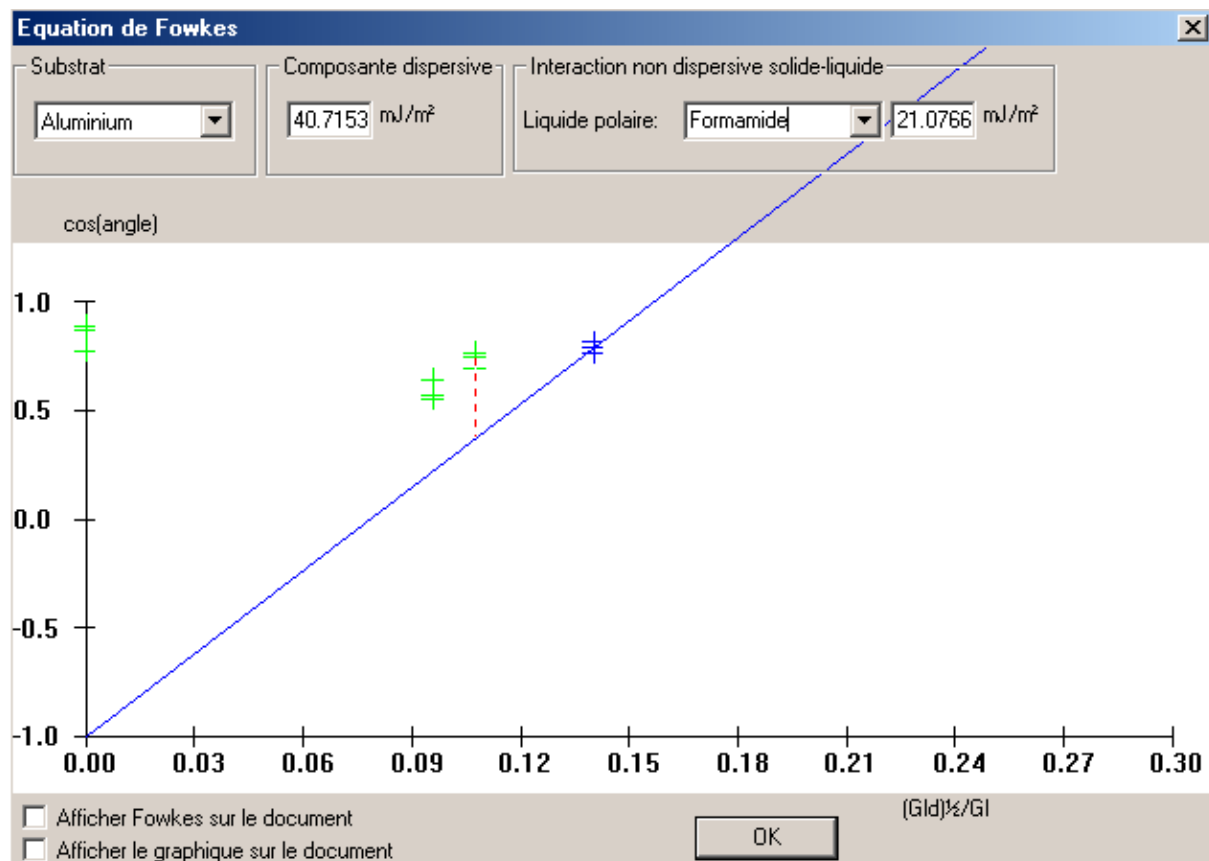


Figure 10-20: Drop test results by Fowkes for Aluminum 2017 with abrasion (80) treatment

10.6.3 Aluminum 2017 – Abrasion (100) treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|-----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | Aluminium | 54.6 | 54.3 | 54.5 | 0.581 | marul |
| 2 | Glycérol | Aluminium | 54.8 | 55.9 | 55.4 | 0.569 | marul |
| 3 | Glycérol | Aluminium | 52.7 | 50.8 | 51.8 | 0.619 | marul |
| 4 | Formamide | Aluminium | 48.3 | 43.6 | 46.0 | 0.695 | marul |
| 5 | Formamide | Aluminium | 43.8 | 40.5 | 42.2 | 0.741 | marul |
| 6 | Formamide | Aluminium | 45.5 | 44.4 | 45.0 | 0.708 | marul |
| 7 | Diiodométhane | Aluminium | 37.2 | 37.2 | 37.2 | 0.797 | marul |
| 8 | Diiodométhane | Aluminium | 35.6 | 34.5 | 35.1 | 0.819 | marul |
| 9 | Diiodométhane | Aluminium | 32.7 | 31.0 | 31.9 | 0.849 | marul |
| 10 | D M S O | Aluminium | 32.8 | 32.1 | 32.5 | 0.844 | marul |
| 11 | D M S O | Aluminium | 28.7 | 29.5 | 29.1 | 0.874 | marul |
| 12 | D M S O | Aluminium | 32.9 | 35.0 | 34.0 | 0.830 | marul |

Table 10-41: Complete table of drop test results for Aluminum 2017 with abrasion (100) treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|-----------|-------|----------|-----------|
| 1 | Glycérol | Aluminium | 53.9 | 3 | 1.53 |
| 2 | Formamide | Aluminium | 44.3 | 3 | 1.61 |
| 3 | Diiodométhane | Aluminium | 34.7 | 3 | 2.20 |
| 4 | D M S O | Aluminium | 31.8 | 3 | 2.03 |

Table 10-42: Sum of abrasion (100) treatment Aluminum 2017

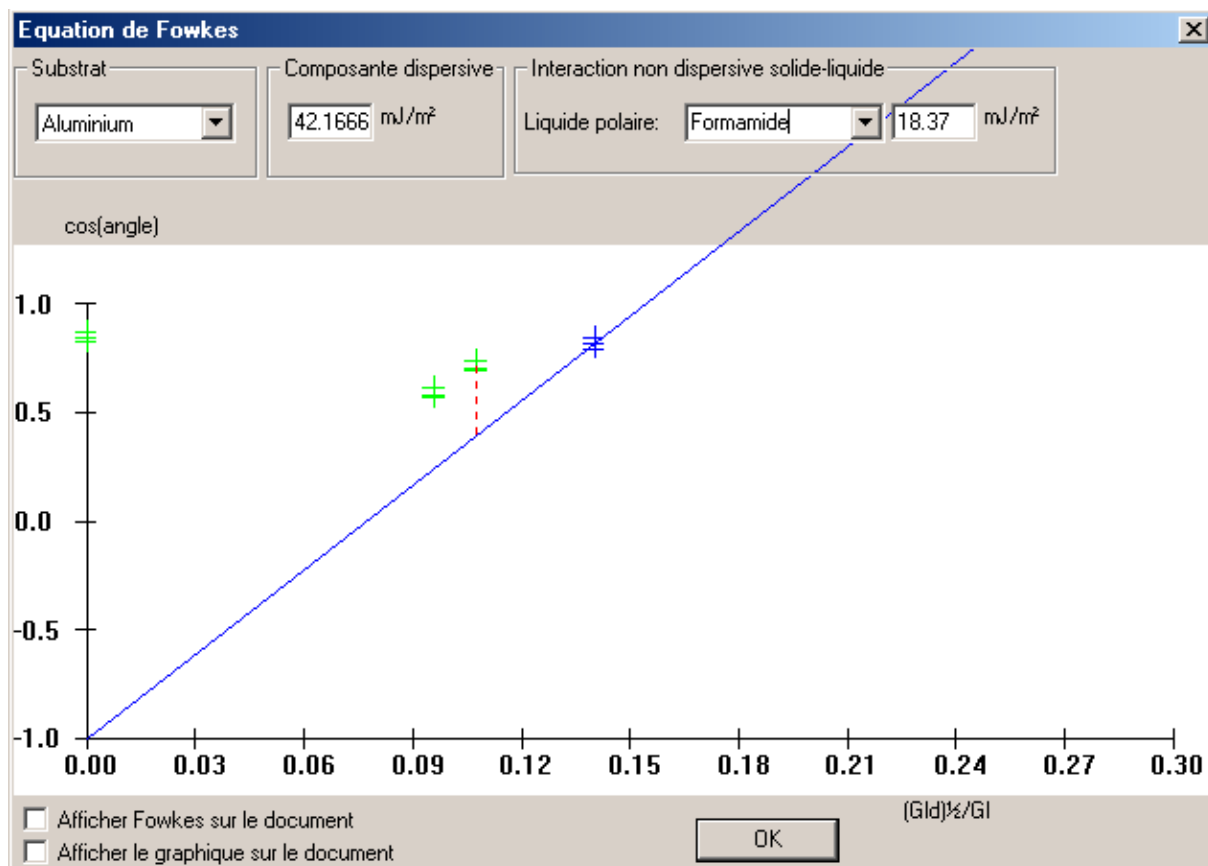


Figure 10-21: Drop test results by Fowkes for Aluminum 2017 with abrasion (100) treatment

10.6.4 Aluminum 2017 – Chemical treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|-----------|-------|-------|-------|-------|-------|
| 1 | DMSO | Aluminium | 12.0 | 12.1 | 12.1 | 0.978 | manul |
| 2 | DMSO | Aluminium | 9.5 | 9.1 | 9.3 | 0.987 | manul |
| 3 | DMSO | Aluminium | 6.5 | 7.0 | 6.8 | 0.993 | manul |
| 4 | Glycérol | Aluminium | 27.7 | 30.5 | 29.1 | 0.874 | manul |
| 5 | Glycérol | Aluminium | 27.7 | 32.2 | 30.0 | 0.866 | manul |
| 6 | Glycérol | Aluminium | 26.2 | 24.8 | 25.5 | 0.903 | manul |
| 7 | Formamide | Aluminium | 27.5 | 26.6 | 27.1 | 0.891 | manul |
| 8 | Formamide | Aluminium | 23.2 | 23.4 | 23.3 | 0.918 | manul |
| 9 | Formamide | Aluminium | 23.7 | 24.9 | 24.3 | 0.911 | manul |
| 10 | Diiodométhane | Aluminium | 25.0 | 27.7 | 26.4 | 0.896 | manul |
| 11 | Diiodométhane | Aluminium | 29.8 | 33.1 | 31.5 | 0.853 | manul |
| 12 | Diiodométhane | Aluminium | 22.5 | 22.1 | 22.3 | 0.925 | manul |

Table 10-43: Complete table of drop test results Aluminum 2017 with chemical treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|-----------|-------|----------|-----------|
| 1 | DMSO | Aluminium | 9.4 | 3 | 2.16 |
| 2 | Glycérol | Aluminium | 28.2 | 3 | 1.93 |
| 3 | Formamide | Aluminium | 24.9 | 3 | 1.59 |
| 4 | Diiodométhane | Aluminium | 26.7 | 3 | 3.74 |

Table 10-44: Sum of chemical treatment for Aluminum 2017

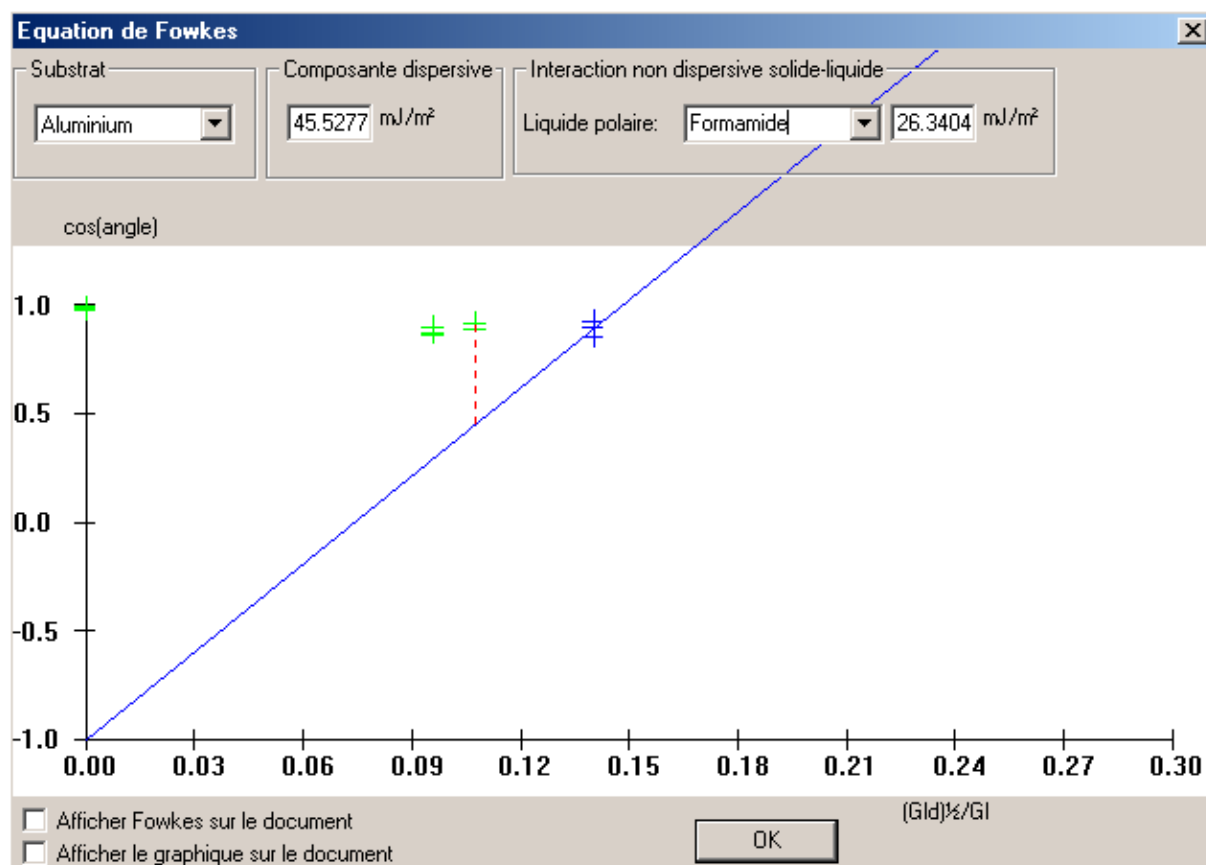


Figure 10-22: Drop test results by Fowkes method for Aluminum 2017 with chemical treatment

10.6.5 Aluminum 2017– Plasma treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|-----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | aluminium | 32.7 | 34.1 | 33.4 | 0.835 | manul |
| 2 | Glycérol | aluminium | 33.4 | 32.1 | 32.8 | 0.841 | manul |
| 3 | Glycérol | aluminium | 43.0 | 40.3 | 41.7 | 0.747 | manul |
| 4 | Formamide | aluminium | 18.2 | 17.2 | 17.7 | 0.953 | manul |
| 5 | Formamide | aluminium | 17.1 | 15.7 | 16.4 | 0.959 | manul |
| 6 | Formamide | aluminium | 19.7 | 18.0 | 18.9 | 0.946 | manul |
| 7 | DMSO | aluminium | 9.5 | 9.6 | 9.6 | 0.986 | manul |
| 8 | DMSO | aluminium | 12.0 | 11.4 | 11.7 | 0.979 | manul |
| 9 | DMSO | aluminium | 6.5 | 6.4 | 6.5 | 0.994 | manul |
| 10 | Diiodométhane | aluminium | 19.7 | 19.0 | 19.4 | 0.944 | manul |
| 11 | Diiodométhane | aluminium | 21.7 | 21.7 | 21.7 | 0.929 | manul |
| 12 | Diiodométhane | aluminium | 20.1 | 20.1 | 20.1 | 0.939 | manul |

Table 10-45: Complete table of drop test results for Aluminum 2017 with plasma treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|-----------|-------|----------|-----------|
| 1 | Glycérol | aluminium | 35.9 | 3 | 4.05 |
| 2 | Formamide | aluminium | 17.7 | 3 | 1.00 |
| 3 | DMSO | aluminium | 9.2 | 3 | 2.15 |
| 4 | Diiodométhane | aluminium | 20.4 | 3 | 0.98 |

Table 10-46: Sum of plasma treatment for Aluminum 2017

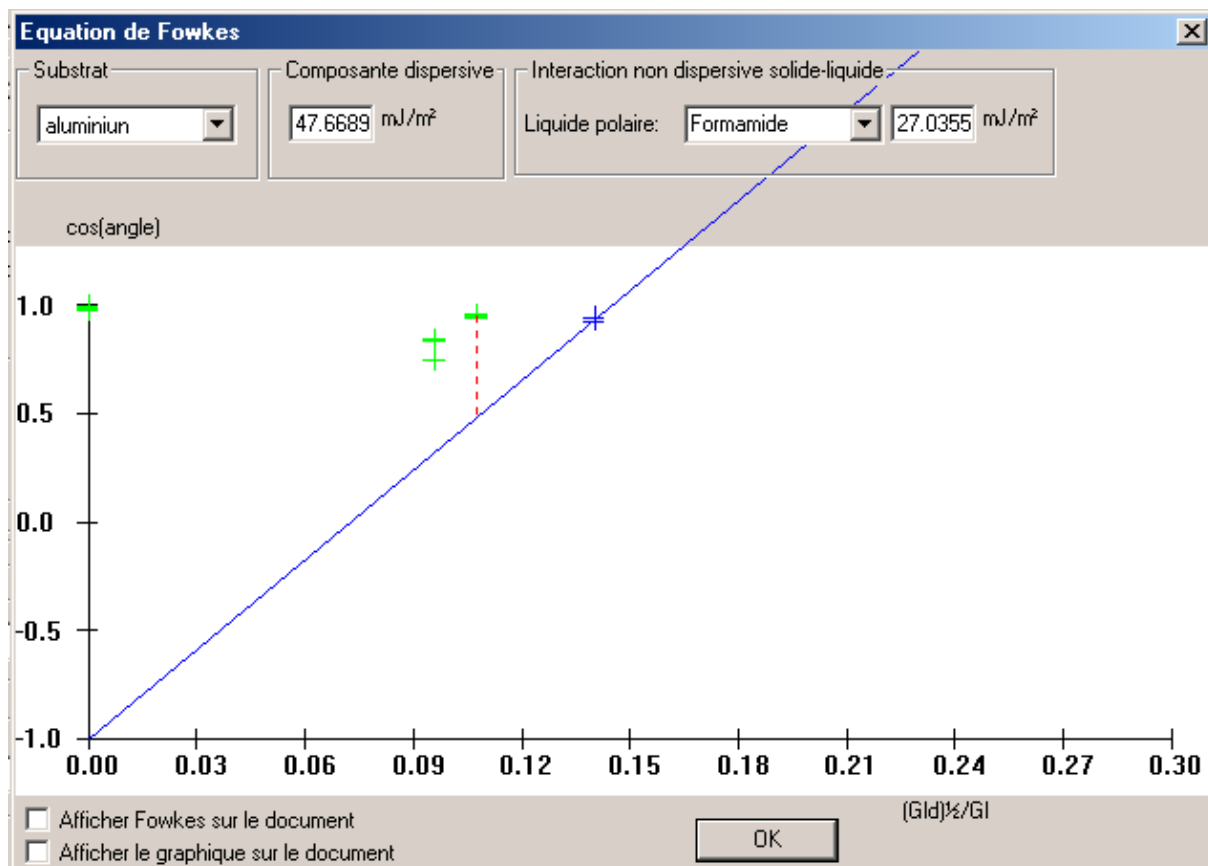


Figure 10-23: Drop test results by Fowkes method for Aluminum 2017 with plasma treatment

10.7 Appendix 7 – Stainless steel 316L drop test results

10.7.1 Stainless steel 316L– Without treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | C os | M ode |
|-----|----------------|-----------|-------|-------|--------|-------|---------|
| 1 | Glycérol | St. Steel | 61.8 | 59.9 | 60.9 | 0.487 | m anual |
| 2 | Glycérol | St. Steel | 57.0 | 54.2 | 55.6 | 0.565 | m anual |
| 3 | Glycérol | St. Steel | 69.4 | 67.1 | 68.3 | 0.371 | m anual |
| 4 | Form amide | St. Steel | 48.6 | 47.0 | 47.8 | 0.672 | m anual |
| 5 | Form amide | St. Steel | 51.1 | 50.6 | 50.9 | 0.631 | m anual |
| 6 | Form amide | St. Steel | 54.5 | 50.9 | 52.7 | 0.606 | m anual |
| 7 | Diiodom éthane | St. Steel | 40.0 | 38.1 | 39.1 | 0.777 | m anual |
| 8 | Diiodom éthane | St. Steel | 40.4 | 38.9 | 39.7 | 0.770 | m anual |
| 9 | Diiodom éthane | St. Steel | 40.9 | 38.3 | 39.6 | 0.771 | m anual |
| 10 | D M S O | St. Steel | 34.0 | 30.0 | 32.0 | 0.848 | m anual |
| 11 | D M S O | St. Steel | 37.2 | 35.7 | 36.5 | 0.804 | m anual |
| 12 | D M S O | St. Steel | 43.3 | 40.7 | 42.0 | 0.743 | m anual |

Table 10-47: Complete table of drop test results for Stainless steel 316L without treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecartype |
|-----|----------------|-----------|--------|----------|----------|
| 1 | Glycérol | St. Steel | 61.6 | 3 | 5.19 |
| 2 | Form amide | St. Steel | 50.5 | 3 | 2.02 |
| 3 | Diiodom éthane | St. Steel | 39.4 | 3 | 0.27 |
| 4 | D M S O | St. Steel | 36.8 | 3 | 4.09 |

Table 10-48: Sum of initial drop test for Stainless steel 316L without treatment

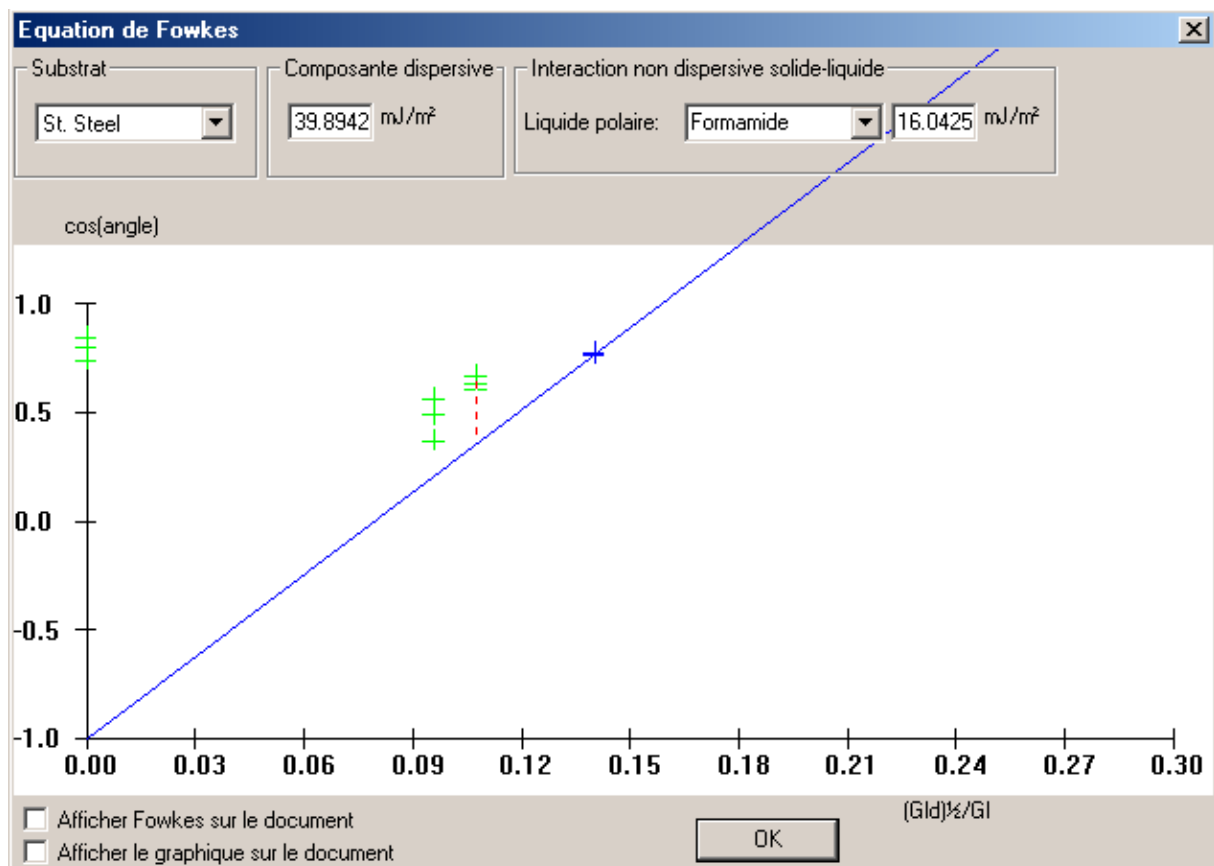


Figure 10-24: Drop test results by Fowkes method for Stainless steel 316L without treatment

10.7.2 Stainless steel 316L – Abrasion (80) treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|---------------|----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | St Steel | 54.7 | 55.9 | 55.3 | 0.569 | manul |
| 2 | Glycérol | St Steel | 56.1 | 57.4 | 56.8 | 0.548 | manul |
| 3 | Glycérol | St Steel | 52.3 | 53.9 | 53.1 | 0.600 | manul |
| 4 | Formamide | St Steel | 46.2 | 49.8 | 48.0 | 0.669 | manul |
| 5 | Formamide | St Steel | 48.1 | 47.4 | 47.8 | 0.672 | manul |
| 6 | Formamide | St Steel | 60.3 | 64.8 | 62.6 | 0.461 | manul |
| 7 | Diiodométhane | St Steel | 41.6 | 42.1 | 41.9 | 0.745 | manul |
| 8 | Diiodométhane | St Steel | 41.4 | 41.4 | 41.4 | 0.750 | manul |
| 9 | Diiodométhane | St Steel | 37.0 | 37.5 | 37.3 | 0.796 | manul |
| 10 | D M S O | St Steel | 35.7 | 32.2 | 34.0 | 0.830 | manul |
| 11 | D M S O | St Steel | 34.2 | 31.3 | 32.8 | 0.841 | manul |
| 12 | D M S O | St Steel | 32.1 | 31.8 | 32.0 | 0.849 | manul |

Table 10-49: Complete table of drop test for Stainless steel 316L with abrasion (80) treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|---------------|----------|-------|----------|-----------|
| 1 | Glycérol | St Steel | 55.1 | 3 | 1.50 |
| 2 | Formamide | St Steel | 52.8 | 3 | 6.92 |
| 3 | Diiodométhane | St Steel | 40.2 | 3 | 2.07 |
| 4 | D M S O | St Steel | 32.9 | 3 | 0.82 |

Table 10-50: Sum of abrasion (80) treatment Stainless steel 316L

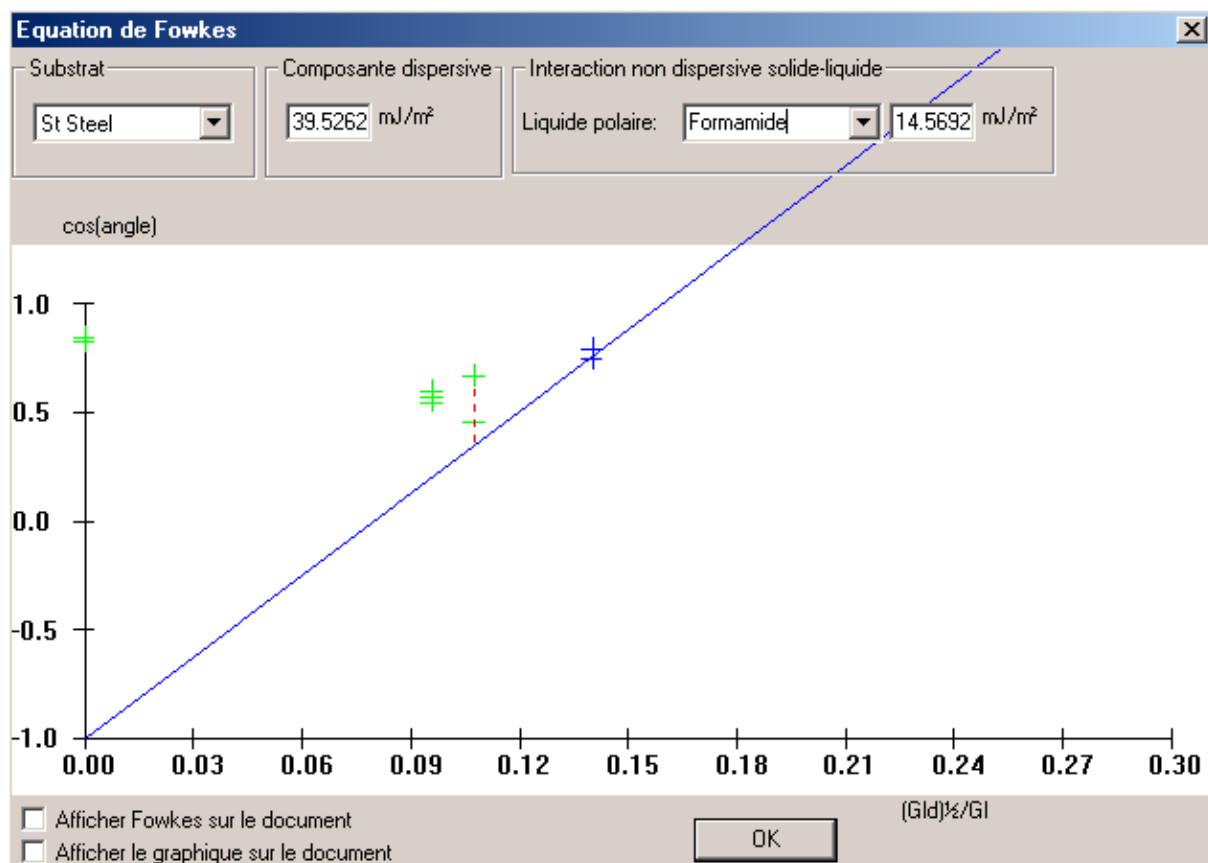


Figure 10-25: Drop test results by Fowkes for Stainless steel 316L with abrasion (80) treatment

10.7.3 Stainless steel 316L – Abrasion (100) treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | C os | Mode |
|-----|---------------|-----------|-------|-------|--------|-------|-------|
| 1 | Glycérol | St. Steel | 47.8 | 45.0 | 46.4 | 0.690 | marul |
| 2 | Glycérol | St. Steel | 44.6 | 45.7 | 45.2 | 0.705 | marul |
| 3 | Glycérol | St. Steel | 47.3 | 47.5 | 47.4 | 0.677 | marul |
| 4 | Formamide | St. Steel | 39.5 | 39.5 | 39.5 | 0.772 | marul |
| 5 | Formamide | St. Steel | 41.8 | 38.8 | 40.3 | 0.763 | marul |
| 6 | Formamide | St. Steel | 40.8 | 38.4 | 39.6 | 0.771 | marul |
| 7 | Diiodométhane | St. Steel | 29.5 | 30.7 | 30.1 | 0.865 | marul |
| 8 | Diiodométhane | St. Steel | 40.7 | 38.0 | 39.4 | 0.773 | marul |
| 9 | Diiodométhane | St. Steel | 36.6 | 38.8 | 37.7 | 0.791 | marul |
| 10 | D M S O | St. Steel | 25.7 | 24.1 | 24.9 | 0.907 | marul |
| 11 | D M S O | St. Steel | 23.5 | 22.8 | 23.2 | 0.919 | marul |
| 12 | D M S O | St. Steel | 23.8 | 24.8 | 24.3 | 0.911 | marul |

Table 10-51: Complete table of drop test for Stainless steel 316L with abrasion (100) treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|---------------|-----------|--------|----------|-----------|
| 1 | Glycérol | St. Steel | 46.3 | 3 | 0.92 |
| 2 | Formamide | St. Steel | 39.8 | 3 | 0.36 |
| 3 | Diiodométhane | St. Steel | 35.7 | 3 | 4.03 |
| 4 | D M S O | St. Steel | 24.1 | 3 | 0.73 |

Table 10-52: Sum of abrasion (100) treatment for Stainless steel 316L

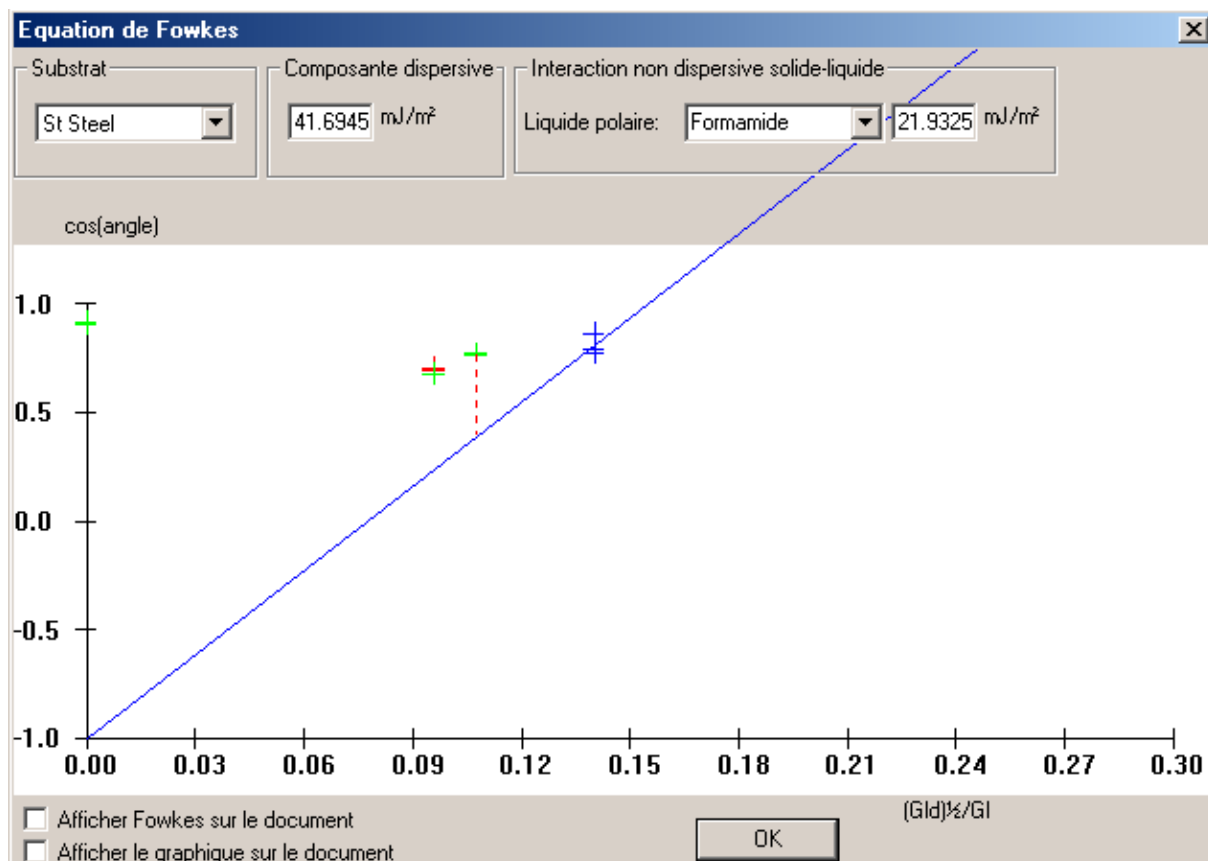


Figure 10-26: Drop test results by Fowkes for Stainless steel 316L with abrasion (100) treatment

10.7.4 Stainless steel 316L – Chemical treatment

| N ° | Liquide | Substrat | A. G. | A. D. | Moyen | Cos | Mode |
|-----|----------------|----------|-------|-------|-------|-------|-------|
| 1 | Glycérol | steel | 7.5 | 7.6 | 7.6 | 0.991 | manul |
| 2 | Glycérol | steel | 8.5 | 8.0 | 8.3 | 0.990 | manul |
| 3 | Glycérol | steel | 10.9 | 10.4 | 10.7 | 0.983 | manul |
| 4 | Formamide | steel | 8.1 | 8.2 | 8.2 | 0.990 | manul |
| 5 | Formamide | steel | 6.2 | 5.4 | 5.8 | 0.995 | manul |
| 6 | Formamide | steel | 10.4 | 9.1 | 9.8 | 0.986 | manul |
| 7 | Diiodom éthane | steel | 9.2 | 8.4 | 8.8 | 0.988 | manul |
| 8 | Diiodom éthane | steel | 11.7 | 10.8 | 11.3 | 0.981 | manul |
| 9 | Diiodom éthane | steel | 10.3 | 10.1 | 10.2 | 0.984 | manul |
| 10 | DMSO | steel | 1.8 | 1.6 | 1.7 | 1.000 | manul |
| 11 | DMSO | steel | 1.7 | 1.7 | 1.7 | 1.000 | manul |
| 12 | DMSO | steel | 1.6 | 1.4 | 1.5 | 1.000 | manul |

Table 10-53: Complete table of drop test results for Stainless steel 316L with chemical treatment

| N ° | Liquide | Substrat | Moyen | Nb. Mes. | Ecarttype |
|-----|----------------|----------|-------|----------|-----------|
| 1 | Glycérol | steel | 8.8 | 3 | 1.33 |
| 2 | Formamide | steel | 7.9 | 3 | 1.62 |
| 3 | Diiodom éthane | steel | 10.1 | 3 | 1.00 |
| 4 | DMSO | steel | 1.6 | 3 | 0.09 |

Table 10-54: Sum of chemical treatment for Stainless steel 316L

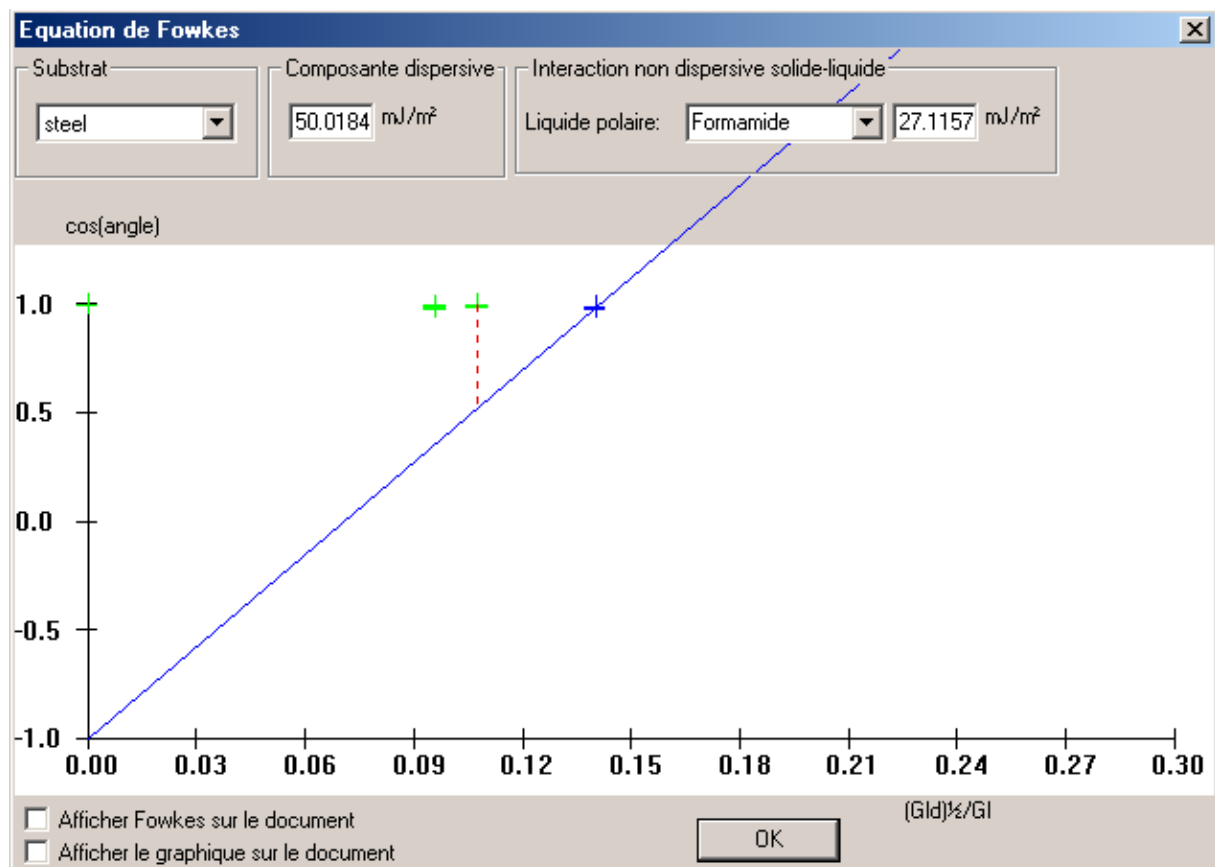


Figure 10-27: Drop test results by Fowkes method for Stainless steel 316L with chemical treatment

10.7.5 Stainless steel 316L – Plasma treatment

| N ° | Liquide | Substrat | A. G. | A. D. | M oyen | Cos | Mode |
|-----|----------------|---------------|-------|-------|--------|-------|--------|
| 1 | Glycérol | | 18.7 | 20.4 | 19.6 | 0.942 | m anul |
| 2 | Glycérol | | 22.9 | 24.2 | 23.6 | 0.917 | m anul |
| 3 | Glycérol | | 22.7 | 24.7 | 23.7 | 0.916 | m anul |
| 4 | D M S O | | 4.4 | 4.3 | 4.4 | 0.997 | m anul |
| 5 | D M S O | | 0.6 | 0.6 | 0.6 | 1.000 | m anul |
| 6 | D M S O | | 1.7 | 1.6 | 1.7 | 1.000 | m anul |
| 7 | Form amide | | 17.1 | 17.8 | 17.5 | 0.954 | m anul |
| 8 | Form amide | | 15.6 | 16.7 | 16.2 | 0.961 | m anul |
| 9 | Form amide | | 16.2 | 17.4 | 16.8 | 0.957 | m anul |
| 10 | Diiodom éthane | stainlessstee | 13.4 | 14.8 | 14.1 | 0.970 | m anul |
| 11 | Diiodom éthane | stainlessstee | 15.0 | 16.0 | 15.5 | 0.964 | m anul |
| 12 | Diiodom éthane | stainlessstee | 15.0 | 15.8 | 15.4 | 0.964 | m anul |

Table 10-55: Complete table of drop test results for Stainless steel 316L with plasma treatment

| N ° | Liquide | Substrat | M oyen | Nb. Mes. | Ecarttype |
|-----|----------------|---------------|--------|----------|-----------|
| 1 | Glycérol | | 22.3 | 3 | 1.92 |
| 2 | D M S O | | 2.2 | 3 | 1.58 |
| 3 | Form amide | | 16.8 | 3 | 0.53 |
| 4 | Diiodom éthane | stainlessstee | 15.0 | 3 | 0.64 |

Table 10-56: Sum of plasma treatment for Stainless steel 316L

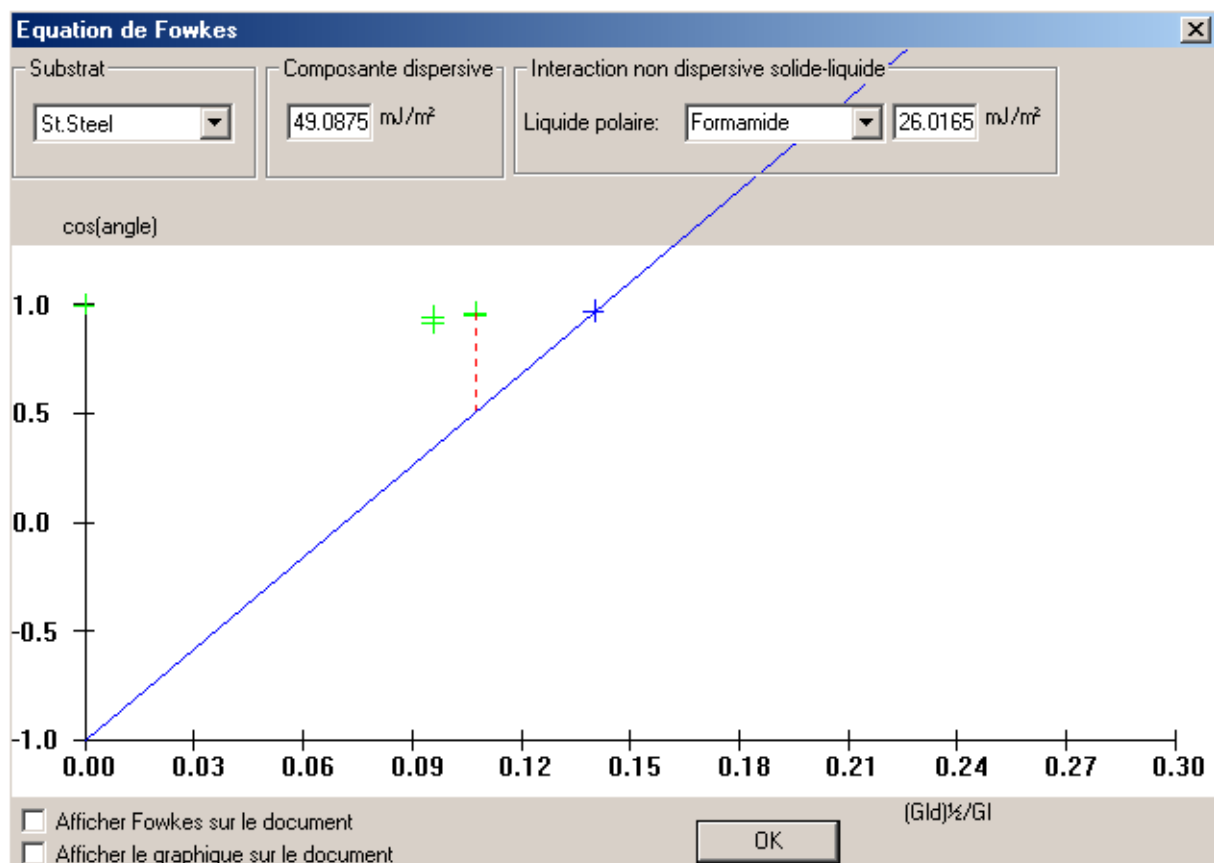


Figure 10-28: Drop test results by Fowkes method for Stainless steel 316L with plasma treatment